HEAT TRANSFER ENHANCEMENT OF NANOFLUID USING RAW, CHEMICALLY MODIFIED AND IMPREGNATED CARBON NANOTUBES

BY

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Dedication to my beloved parents, my wife, my brother, my sisters and my daughter

Lujain

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Q : Heat transfer rate in tube.

m_n : Mass flow rate for nanofluids.

Cp_n : Specific heat capacity of nanofluids.

$T_{n,in}$: Tube inlet temperature.

$T_{n,out}$: Tube outlet temperature.

n : Subscript for nanofluid.

Re : Reynolds's number.

Nu : Nussle's number.

ΔP : Pressure Drop.

DSC : Differential Scanning Calorimetry.

SEM : Scanning ElectronMicroscopy.

TGA : Thermogravimetric Analysis.

FTIR : Fourier Transfer Infrared.

MWCNT : Multi-Walled Carbon Nanotubes.

PEG : Polyethylene glycol.

ABSTRACT

Full Name : [Abdallah Darweesh Omar Manasrah]

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In this present study the heat capacity, viscosity, enhancement of heat transfer and pressure drop of the nanofluid with unmodified and modified carbon nanotubes with metal oxide nanoparticles (iron oxide, copper oxide and aluminum oxide) and functionalized with polyethylene glycol (PEG) functional groups were investigated. Tube and shell heat exchanger was used to conduct the experiments of the heat transfer and the pressure drop of the nanofluid at 0.01 wt%, 0.05 wt % and 0.1 wt % of unmodified and modified CNTs. The carbon nanotubes surfaces were doped with metal oxide nanoparticles at 1 wt % and 10 wt % loading and functionalize with PEG using wet impregnation technique and esterification method. The raw and modified CNTs were characterized by different techniques such as Scanning Electron Microscopy (FE-SEM),Transmission Electron Microscopy (HR-TEM), Fourier Transform Infrared Spectroscopy (FTIR) and Thermo Gravimetric Analysis (TGA). The nanofluids were prepared by mixing fixed amount of unmodified and modified CNTs with water as base

fluid and it was sonicated for 1 hr to get a homogenous solution. The specific heat capacities (Cp) of the nanofluids were measured by Differential Scanning Calorimetric (DSC) technique. The results show that the specific heat capacity of unmodified and modified CNTs mixed with water is significantly higher than pure water. While the heat transfer of the nanofluid increased sharply by increasing the dosage of the unmodified and modified CNTs. It was noted that, the metal oxide nanoparticles and the PEG functional groups enhance the dispersion of the CNTs and increased their heat capacities and the heat transfer of the fluid. Furthermore there is no effect neither of the CNTs nor modified CNTs on the pressure drop of the system.

ملخص الرسالة

الاسم الكامل: عبدالله درويش عمر مناصره

عنوان الرسالة:تحسين انتقال الحرارة في المبادلات الحراريه باستخدام السوائل النانونيه المعدله فيزائيا وكيميائيا

التخصص: هندسه كيميائيه

تاريخ الدرجة العلمية: حزيران 2014

إن هذا البحث يهدف إلى دارسة الخصائص الحراريه للموائع النانويه وإستخدامها في عملية إنتقال الحراره في المبادلات الحراريه.

الموائع النانونيه هي موائع تم تصنعيها حديثاً نتيجه للتطور الذي حصل في الأجهزه المستخدمه في تشيخيص المواد التي تحتوي على ابعاد في مقياس النانو . حيث يتم تحضير هذه الموائع عن طريق غمس المواد النانويه في تلك الموائع التقليديه التي استخدمت سابقا في عمليات الانتقال الحراري كالماء والزيوت، ونتيجه لذلك يتم الحصول على مزيج متجانس وذات موصليه حراريه عاليه.

في هذا البحث، وبعد تحليل عينات الأنانيب الكربونيه بإستخدام جهاز المسح السطحي، تم إنتاج موائع نانونيه من خلالنثر أو غمسالأنابيب الكربونية ذات المواصفات العاليه في داخل الماء.ومن ثم فحص خصائص النقل الحراري للموائع كالحرارة النوعيه والموصليه الحرايه واللزوجة.حيث تبين أن الحراره النوعيه للموائع النانونيه أعلى من الموائع التقليديه بمقدار 12%.

بالأضافه الى ذلك، تم إستخدام هذه الموائع في المبادل الحراري لدراسة تأثير المواد النانوية على التوصيل الحراري للمبادل الحراري. ثلاث عوامل رئيسة تم دراستها وهي كمية المواد النانونية ومعدل تدفق المانع ودرجة حرارة المبادل الحراري وذلك لإيجاد أفضل العوامل التي تعطي أعلى توصيل حراري في المبادل الحراري. حيث تبين أن

معدل التوصيل الحراري ازداد بمقدر 16% مقارنةً مع الماء عند استخدام نسبه قليل جدا من الماده النانونيه لايزيد وزنها عن 0.1%. بالاضافه الى ذلك حيث وجد ان فرق الضغط بين أنابيب المبادل الحراري لم يتأثر بوجود الموائع النانونيه.

CHAPTER ONE

INTRODUCTION

1.1 Background

Heat transfer is an important area of study in thermal engineering. Selection of a suitable heat transfer fluid for heat dissipation is an important consideration in the thermal design of heat exchangers. Heat transfer fluid is one of the critical parameters which affects the cost and size of heat exchanger systems. Conventional fluids like water and oils have limited heat transfer potentialities. The need for development of new classes of fluids with enhanced heat transfer capabilities is sensed by different research groups around the world. The advances in nanotechnology have made it possible to manufacture metal and metal oxide particles on a nano-dimensional scale. Nano particles are considered to be new generation material having potential applications in the heat transfer area.

Any host liquid which contains nanoparticles in a suspended state is known as a nanofluid. Nanofluids are two phase fluids of solid-liquid mixtures and are considered to be new generation heat transfer fluids. In the recent past nanofluids have emerged as promising thermo fluids for heat transfer applications. The thermal conductivity property of nanofluids is expected to be greater than that of the base liquids. The practice of adding micron size particles to traditional heat transfer fluids has existed since the time of [1]. But two phase suspensions of micro particles produce sedimentation obstructions to smooth fluid flow due to channel clogging and cause erosion of tube materials.

Nanofluids, on the other hand, offer many advantages over single phase pure fluids and suspensions with micro particles. The problems of particle sedimentation due to gravity, clogging of micro channel passages, and erosion of tube material are minimized to a great extent when nanofluids are used in heat exchangers. Besides, nanofluids form stable suspensions with uniform dispersion of nanoparticles in the host fluid. Thermophysical properties of single phase heat transfer fluids such as oils, water and glycols are well established and are available in literature and handbooks. However, the thermophysical properties of two phase nanofluids have not explored extensively. An accurate measurement of the properties of nanofluids is a prerequisite for the determination of heat transfer coefficients of nanofluids. The suitability of a particular nanofluid in a heat transfer application is then judged based on its heat transfer performance. Nanofluids are considered to be an alternate and new generation of liquids for heat energy transport and can be employed as heat transfer fluids in heat exchangers in place of pure single phase fluids. The applications of nanofluids for heat transfer include radiators in automobiles, components in chemical engineering and process industries, solar water heaters, refrigeration units, and in the cooling of electronics devices. The main objective of obtaining heat transfer enhancement using nanofluids is to accommodate high heat fluxes and, hence, to reduce the cost and size of heat exchangers which, in turn, results in the conservation of energy and material.

Over the last several years, extensive research has been carried out in order to develop heat transfer enhancement methods. Generally, many additives have been used to increase the heat transfer features of base fluids. Therefore, nanofluids may be perfectly suited in actual applications as their use may have little increases in pressure drop, and may

positively change the heat transfer characteristics and transport properties of the fluid. Due to the fine nature of these nano-particles, nanofluids behave as a single phase rather than as a solid-liquid mixture, multiphase.

Since discovering Carbon nanotubes in 1991 a great deal of interest has been generated. Carbon nanotubes have a thermal conductivity at room temperature 3,000 times higher than water and 10,000 times higher than engine oil [2].

The high variations in thermal conductivity between carbon nanotubes and other metals and liquids are shown in Table 1.1.

Table 1.1 : Thermal conductivity of different solids and liquids (Marquis and Chibante 2005) [2].

Material	**Form**	**Thermal Conductivity (W/mK)**
Carbon	Nanotubes	1,800-2000
	Diamond	2,300
	Graphite	110-190
Metallic Solids	Copper	401
	Silver	429
	Aluminum	237
	Nickel	158
Non-Metallic Solids	Silicon	148
	Alumina	40
Metallic Liquids	Sodium	72.3
Nonmetallic liquids	Water	0.613
	Ethylene Glycol	0.253
	Engine oil	0.145

Therefore, fluids containing suspended carbon nanotubes are expected to have significantly greater thermal conductivity compared to common heat transfer fluids. Multi-wall carbon nanotubes (MWNTs) and Single wall carbon nanotubes (SWNTs) have been found to significantly enhance the thermal conductivities of many heat transfer fluids like water, oil, water with ethylene glycol mixtures and antifreeze. Homogenization and sonication were some of the techniques used to achieve good dispersions of CNTs in these fluids by mixing in suitable solvents. CNTs are supposed to contribute the thermal conductivity of heat transfer fluids through Brownian motion and by configuration of a tri dimensional network within the fluids. The primary evaluation of the heat transfer properties of nanofluids such as viscosity, pH and density have the potential to find a place in a variety of applications such as engine cooling systems, heat pumps and oil coolers. Nanofluids are expected to considerably increase the performance of these thermal and lubricating fluids [2].

1.2 Significance of Study

There are many heat transfer fluids commonly in use like ethylene glycol, water and mineral oil that have a role in industrial processes such as heating and cooling, power generation, chemical processes and microelectronics. These fluids have poor heat transfer characteristics compared to solids and this is the main obstacle to high effectiveness and compactness of heat exchangers. The enhancement in specific heat capacity of these fluids is an important method to improve heat transfer characteristics.

1.3 Problem Statement

This research focuses on experimental investigation of heat transfer characterization for CNT-nanofluid in heat exchangers taking into account various parameters such as dosage of CNT, type of metal oxide nanoparticles loaded onto the CNTs and type of surfactant.

1.4 Research Objectives

The main objectives of this work are:

1) To impregnate the surface of CNTs with metal oxide nanoparticles such as copper oxide, iron oxide and aluminum oxide.

2) To functionalize the surface of CNTs with poly ethylene glycol to enhance the dispersion of carbon nanotubes inside the considered fluid (water).

3) To characterize the CNTs as well as the functionalized/impregnated CNTs using Fourier transfer infrared (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) and scanning electron microscopy (SEM) techniques.

4) To study the effect of the modified (functionalized/impregnated) and unmodified CNTs on the properties of the nanofluid such as specific heat capacity.

5) To study the effect of modified and unmodified CNTs dosage and the flow rate of the nanofluid on the thermal efficiency of the heat exchanger.

6) To study the effect of the modified (functionalized/impregnated) and unmodified CNTs on the pressure drop of shell and tube heat exchanger.

CHAPTER TWO

2 LITERATURE REVIEW

The thermophysical properties required for investigation of the convective heat transfer coefficient are thermal conductivity, specific heat capacity, viscosity and density. Properties of single phase fluids are well documented and are available in heat transfer data publications. But on the other hand, the thermophysical properties of two phase fluids are not readily available and need to be measured by conducting experiments. Researchers have elaborated experimental procedures for measurement of nanofluid properties. Earlier studies have reported that nanofluid properties vary with temperature and particle concentration in the base fluid. The thermal conductivities of two phase nanofluids are comparatively higher than those of conventional fluids.

Properties of nanofluids can be altered to make them suitable particularly in heat transfer applications. Conventional heat transfer fluids can be replaced by nanofluids offering many advantages.

Thermal conductivity is one of the significant properties that influence heat transfer in nanofluids. The heat transfer characteristics of nanofluids depend on fluid mass flow rate; type of flow whether it is laminar or turbulent and also whether there is swirl in the flow, created by different inserts.

2.1 Literature Review on Nanofluid Properties

Many thermophysical properties have been investigated for heat transfer fluid characteristics, such as thermal conductivity, specific heat capacity, density, and viscosity. The most important is thermal conductivity, because improvement of the thermal conductivity of nanofluids is a key to the enhancement of its heat transfer characteristics. Consequently, many experiments have been conducted to investigate these properties.

The novel idea that has been conceived is to use nanoparticles in two-phase fluids. This fluid mixture is expected to produce high thermal conductivity. Nano particles of metals and metal oxides dispersed in any conventional heat transfer fluids show higher thermal conductivities compared to the thermal conductivities of pure liquids. In the last 100 years, a number of theoretical and experimental studies have been undertaken on the properties of liquid suspensions containing milli or micro sized particles.

Touloukian in 1977 proved experimentally, that at room temperature, the thermal conductivity of Cu is 700 times more than water and 3000 times more than engine oil. Wasp, Kenny et al. in 1977 and Hamilton and Crosser in 1962 have developed a thermal conductivity model for a two-phase mixture based on their theoretical study. Masuda, Ebata et al. in 1993 studied the possibility of altering the properties of conventional heat transfer fluids by suspending submicron particles of water based Al_2O_3 and TiO_2 and reported that the enhancement in the thermal conductivities were about 32 % and 11%, respectively for the nanofluids of a 4.3% volume concentration.

In 1995 Choi is the first researcher who worked on nanoparticles at the Argonne National Laboratory, USA. He demonstrated that the thermal conductivity of nanofluids increased

compared to the base fluid. Eastman, Choi et al. in 1997 observed that Al_2O_3 and CuO nanoparticles have good dispersion properties with stable suspensions in oil, ethylene glycol and water.

In 1999 Wang, Xu et al. employed a steady state parallel plate method for measuring the thermal conductivity of nanofluids. They tested two types of nanoparticles, CuO and Al_2O_3, dispersed in water, ethylene glycol, and engine oil. Experimental results indicated higher thermal conductivities in fluid mixtures than those of the base fluids and greater measured thermal conductivity values for nanofluids.

The maximum enhancement of thermal conductivity was described in 2001 by Choi and Zhang. A nanofluid was prepared by suspension of MWCNTs in engine oil. They found an approximate 160% enhance in thermal conductivity for a 1 vol.% of the nanotubes by using a transient hotwire method. Das in 2003employed a temperature oscillation technique to estimate the thermal conductivity of water based Al_2O_3 and CuO nanofluids at different temperatures and observed a 200%to 400% increase in the thermal conductivity of nanofluids when the temperature ranged from 21^{o}C to 51^{o}C.

Assael in 2004 investigated the thermal conductivity of a MWCNT suspension in water employed with a dispersant agent like Sodium dodecyl sulfate (SDS) and using the transient hotwire technique. They found that an approximate 38% enhancement in the thermal conductivity for 0.6 vol. % concentration of MWCNTs.

In 2005 Assael, Metaxa et al. measured thermal conductivity at ambient temperature for MWCNT as well as DWCNTs suspended in water. Their results displayed a 34% increase

in thermal conductivity for 0.6 vol. % fractions of MWCNTS whereas the results displayed a 7.6 % increase for a 1 % volume fraction of double walled CNTs.

Xue and Xu in 2005 developed a thermal conductivity model for CuO-water and CuO-Ethylene glycol nanofluids taking into account the thermal conductivity of the liquids and solids, volume fraction, particle size and interfacial properties. Koo and Kleinstreuer in 2005studied the conduction-convection heat transfer features of water-ethylene-CuO nanofluids in micro channels and developed new models for thermal conductivity and viscosity including the effect of viscous dissipation. Murshed, Leong in 2005used cylindrical and spherical shaped TiO_2 nanoparticles in water and measured the thermal conductivities by applying the hot wire method. Their results revealed that the thermal conductivity enhanced by increasing the concentration of nanoparticles. The particle shape and size also had a bearing on improvement of thermal conductivity. They also characterized viscosity and pH values of the nanofluids.

Liu, Lin et al in 2006 produced Cu nanoparticles of around 50–100 nm in diameter by a chemical reduction method and nanofluid prepared without adding a surfactant. They observed a 12.5% increase in the thermal conductivity for a 1% volume fraction of CNT suspension in ethylene glycol whereas a 30% enhancement for a 2% volume fraction of CNT suspension in synthetic oil.

Liu, Ching-Cheng Lin et al. in 2005 investigated thermal conductivities of Carbon Nano Tubes (CNT), CuO, SiO_2 suspended in water by using the transient hot-wire method and reported an 11.3% improvement in the thermal conductivity of CNT-water nanofluids with 0.01% volume concentration. The measured thermal conductivity was relatively higher than the thermal conductivity calculated using the Hamilton–Crosser conductivity

model. Beck, Sun et al. in 2007 measured the thermal conductivity of alumina suspended in ethylene nanofluids in the temperature range of 298 to 411K using the transient hot wire method. Higher thermal conductivities were reported for all concentrations of nanofluids compared to the base liquid.

Velve Casquillas, Le Berre et al. in 2007 conducted experiments on thermal conductivity properties of ethylene glycol based nanofluids of carbon nanotubes at the droplet level and found that nanotube concentration has a strong effect on thermal conductivity. In 2007 Chen, Ding et al. studied and measured shear viscosity of ethylene glycol-titania nanofluids up to 8% percent on a particle weight basis and concluded that the shear viscosity of the nanofluids is a strong function of particle concentration and nanofluid temperature. Hong, Yang et al. in 2005 worked on nanofluids containing carbon nanotubes and Fe_2O_3 particles; he observed that the thermal conductivity of nanofluids can be improved by applying a magnetic field. He reasoned that the Fe_2O_3 particles align in chains formed under an applied magnetic field and connected the nanotubes, which results in enhanced thermal conductivity.

Lee, Hwang et al. in 2008 estimated both thermal conductivities and viscosity properties of Al_2O_3-water nanofluids and observed that both properties increased linearly with an increase in the nanoparticle concentration. Li, Zhu et al. in 2008 investigated the combined effects of pH variation and sodium dodecyl benzene sulfonate as a surfactant on Cu-nanoparticles nanofluids. They observed that the thermal conductivity of Cu-water nanofluids is reliant on the nanoparticle concentration, the pH value of the nanofluid and the concentration of surfactant. For Cu nanoparticles of 0.01 % concentration with an optimum pH value and surfactant, the highest thermal conductivity reported was 10.7 %.

A suitable surfactant and an optimum pH value play a role in improving the thermal conductivity of nanofluids.

Mintsa, Roy et al. in 2009 measured the thermal conductivity of water based alumina and CuO nanofluids. Their results show an increase in the thermal conductivity of nanofluids by increasing the volume concentration and decreasing the size of particle. It is also noted that the relative increase in thermal conductivity was predominant at higher temperatures. Murshed, Leong et al. in 2008 stated that the thermal conductivity of nanofluids is affected by particle shape, size, interfacial layer, and temperature in addition to the particle volume fraction. Zhu, Li et al. in 2009 studied the thermal conductivity and dispersion behavior of Al_2O_3-H_2O nanofluids by varying its pH values and the sodium dodecyl benzene sulphonate concentration. The nanofluids exhibited better dispersion behavior when the surfactant was added in the nanofluid. Lee, Hwang et al. in 2008 measured the effective viscosities and thermal conductivities of low concentration water-Al_2O_3 nanofluids. The nanofluid viscosity decreased with an increase in the temperature. However, the measured thermal conductivity increased linearly with an increase in nanofluid concentration.

Namburu, Kulkarni et al. in 2007 investigated the rheological behavior of CuO nanoparticles suspended in a water-ethylene glycol mixture base fluid by varying the nanoparticle concentrations from 0% to 6.12% in the temperature range from 35^{o}C to 50^{o}C. The nanofluid also exhibited Newtonian behavior in the concentration range tested. For 6.12% volume concentration at 35^{o}C, the viscosity of copper oxide nanofluid is four times greater than the base fluid.

Recently, a new model was described by Geo and Zhou in 2006 using a Maxwell–Garnett model. The model supposes that the thermal conductivity of MWCNT nanofluids with concentration of the carbon nanotubes exhibits non-linear dependence, according to the differential effective medium theory. There are more models for predicting the thermal conductivity of nanofluids, but for all the available results no single theory can conform to the experimental observations.

A recent work was developed by Meibodi et al. in 2010, which they investigated the effects of concentration and size of nanoparticles, types and concentration of surfactants, temperature, pH, and the power of ultrasonication on the thermal conductivity and stability of CNT-water nanofluids. Their results show that it may not be important to have higher values of thermal conductivity for the stability of these nanofluids. . This is probable because the thermal conductivity depends on time immediately after ultrasonication but it became independent at longer times.

Fedele, Colla et al. in 2012 measured the viscosity of nanofluids prepared by water suspended with titanium oxide at different concentration of nanoparticles. Their results presented that the deviations of nanofluids and water viscosities increase as the concentration of TiO_2 is increased. For example, deviation was about 20% at 10 wt. % TiO_2 concentration, whereas at 35 wt. % the deviation was 215 %. For instance, Hojjat, Etemad et al. in 2011 measured the rheological behavior of suspensions of γ-Al_2O_3, TiO_2, and CuO nanoparticles in an aqueous solution of carboxymethyl cellulose at different temperatures. They found that the viscosity of the nanofluid and that of the base fluid are a function of temperature and particle concentration. They observed that the relative apparent viscosity of Al_2O_3 and TiO_2 nanofluids increases with increasing nanoparticle

concentration but the viscosity of CuO nanofluids is practically independent of nanoparticle concentration.

In later research, Moraveji and Razvarz in 2012 investigated the thermal enhancement in pipe using the aluminum oxide nanoparticles into water nanofluids at different operating systems. The concentration of nanofluids was ranged between 0 to 3 wt. %. Their results presented that the thermal efficiency in the heat pipe increased with using nanofluids by reducing thermal resistance.

2.2 Literature on Laminar Flow of Nanofluids Heat Transfer

With the advent of recent advances in nanotechnology, nanomaterial synthesis, processing and characterization are possible and both metallic and non-metallic particles in nano-dimension with controlled structure are being produced. In nanomaterials several parameters like shape, size and rapid interface alter the properties of nanoparticles which are quite different for the same materials in macroscopic dimension. The base liquids containing nanoparticles are known as nanofluids.

Many experimental studies have been initiated by different research groups to study the transport behavior of nanofluids.

Wen and Ding in 2004 investigated the convective heat transfer coefficient of γ-Al_2O_3particles with 27 to 56 nm range size suspended in deionized water under laminar flow conditions and constant wall heat flux in a copper tube. Different concentrations of nanoparticles were used (0.6, 1.0, and 1.6 vol. %). As shown in Figure 2.1, the nanoparticle experimental system involved of a heating and cooling unit, a flow loop, and

a measurement and control unit. The tube is 970 mm long, and is made from copper. . The inside diameter (ID) is 4.5 mm and the outer diameter (OD) is 6.35 mm. To keep heat flux constant a silicon rubber flexible heater was installed. The maximum flow delivery rate was10 L/min. A dispersing agent like sodium dodecylbenzene sulfonate was used in order to stabilize the nanoparticles.

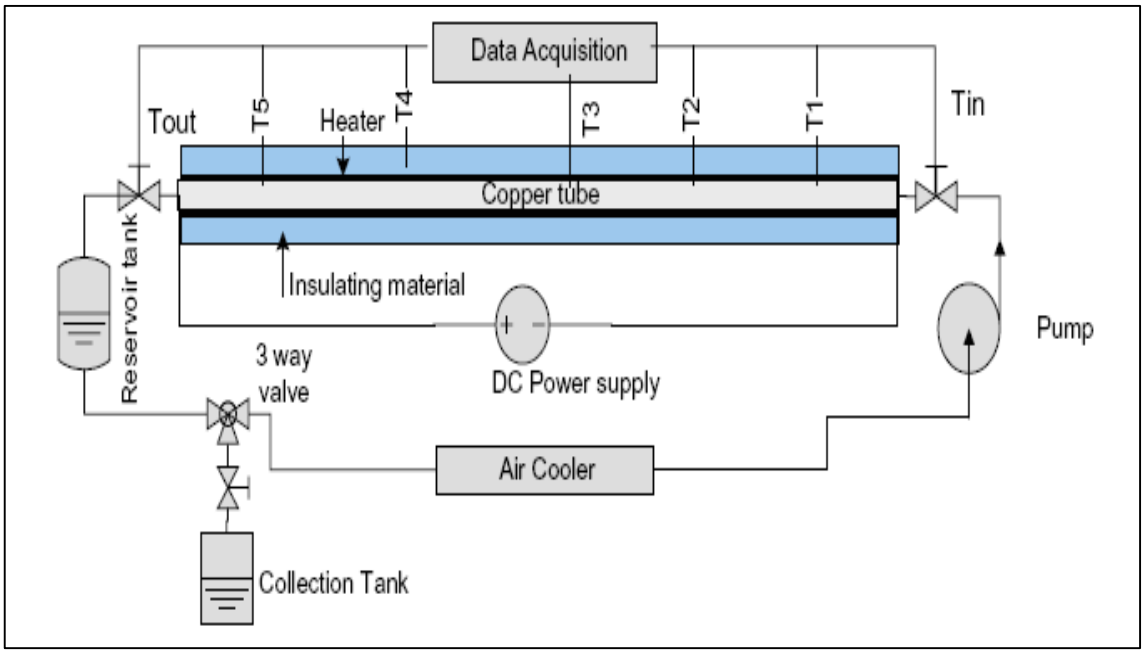

Figure 2:1 Experimental system of Wen and Ding (2004)

An enhancement of convective heat transfer coefficient under laminar flow was shown, and this augmentation also increased by increasing the Reynolds number and concentration of particles. They proposed that this enhancement may be due to migration in particles, which causes a random distribution of thermal conductivity.

Ding, Alias et al. in 2006 studied the effect of MWCNTs on the heat transfer performance of nanofluids using a straight copper tube. The nanofluids were produced by dispersing MWCNTs in distilled water. They found that with increasing concentrations of CNT and nanofluid temperature the thermal conductivity increased as well as the viscosity at a given shear rate. They also found that the highest enhancement of the convective heat transfer coefficient was 350% for 0.5 wt. % of CNT at an 800 Reynolds number. They explained this enhancement by suggesting possible reasons. For example, the CNTs have high aspect ratios, the presence of nanoparticles in the thermal boundary layer may be reduced, and the re-arrangement of particles, and shear-induced thermal conduction enhancement could all be explanations.

In 2005 Yang et al. conducted experimental work which showed the convective heat transfer coefficient in laminar flow in a horizontal tube heat exchanger for nanofluid produced by using graphite nanoparticles with a mean diameter from 1 to 2 μm and with thickness from 20 to 40 nm, dispersed in liquid. Their study focused on the aspect ratio of nanoparticles from different sources, which were about 0.02. The viscosity of nanofluids dramatically increased when adding large aspect ratio particles.

Their experiments aimed at finding the effects of the nanoparticle source, volume fraction, Reynolds number, temperature and kind of conventional fluid on the convective heat transfer coefficient. They performed many experiments by changing the volume flow

rates from 62 to 507 cm^3/min, with a Reynolds number from 5 to110, and fluid temperature from 50 to 70^oC. Figure 2.2., Shows the experimental system of their work.

They observed that the heat transfer characteristics were enhanced by increasing the Reynolds number and the particle concentration with laminar flow. It was shown that there was about a 22% increase in heat transfer coefficient of the nanofluids for 2.5 wt. % compared with the base fluid at 50 ^oC. Many factors may illustrate the enhancement of heat transfer with increased temperature. Firstly, the nanoparticles are rapidly aligned in less viscous fluids, which lead to minimize nanoparticle contact. Secondly the depletion of particles near the wall where fluid phase may lead to reduced thermal conductivity. Different values of heat transfer coefficient were obtained for the same weight fraction of two graphite nanoparticles but with different base fluids. This depends on surface treatment, morphology, and particle shape.

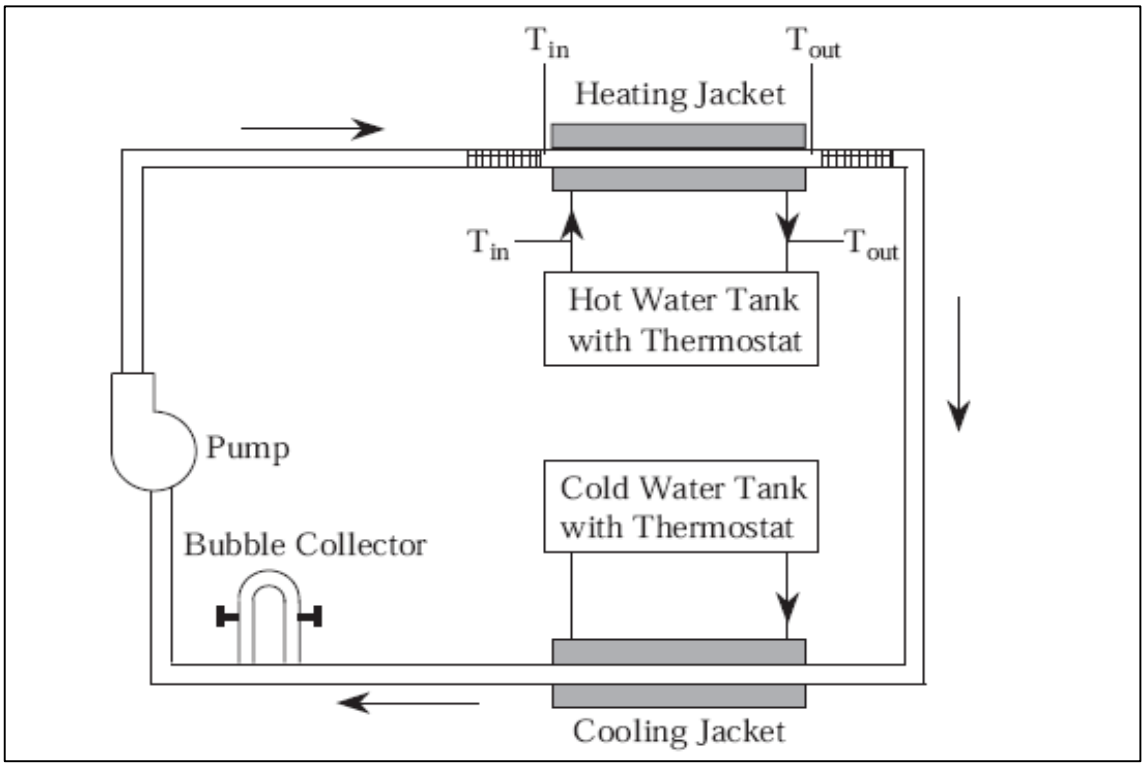

Figure 2:2 The experimental system of Yang et al. (2005)

Heris et al. in 2006 their experimental work on two types of nanofluids (Al_2O_3-water and CuO-water) in a laminar flow regime and subjected to constant wall temperature boundary conditions stated that for both the nanofluids the heat transfer rate increased with increase in the nanoparticle concentration and also with Peclet number. The reasons attributed to enhanced heat transfer are, increased thermal conductivity, random and chaotic movements, and fluctuations and interactions among nanoparticles. Heris, Esfahany et al. in 2006 conducted an experimental study exclusively on Al_2O_3-water nanofluid with 0.2 to 2.5% volume fraction concentrations of nanoparticles under laminar flow and constant wall temperature boundary conditions and observed higher heat transfer coefficients with the nanofluids. It was concluded that, in addition to increased thermal conductivity of nanoparticles, other parameters like chaotic movement of nanoparticles, dispersion, Brownian motion, particle interaction and particle migration may augment heat transfer in the nanofluids.

Asirvatham, Vishal et al. in 2009 investigated the convective heat transfer in laminar flow under steady state conditions of nanofluid flows through a copper tube. The nanofluid was prepared by dispersed copper oxide nanoparticles in deionized water at a 0.003 % volume fraction.

Figure 2.3 shows the experimental setup of their work. It consisted of a copper tube 8 mm in diameter and 1500 mm in length. To avoid any heat leakage ceramic glass wool was used to insulate the copper tube. The tube surface was divided into 17 cm intervals in order to embed eight T-type thermocouples to measure the surface temperature along the whole tube. To measure the pressure drop of nanofluids along the tube, two pressure transducers were installed.

The results displayed that the heat transfer coefficient increased by 8% more than base fluid due to nanoparticles present in the nanofluid. It was also found that the heat transfer coefficient at the entrance region increased, whereas for the axial distance the heat transfer coefficient decreased. According to this study, the major factors that may have enhanced the heat transfer are the particle's chaotic movement and the thermophysical properties of nanofluids.

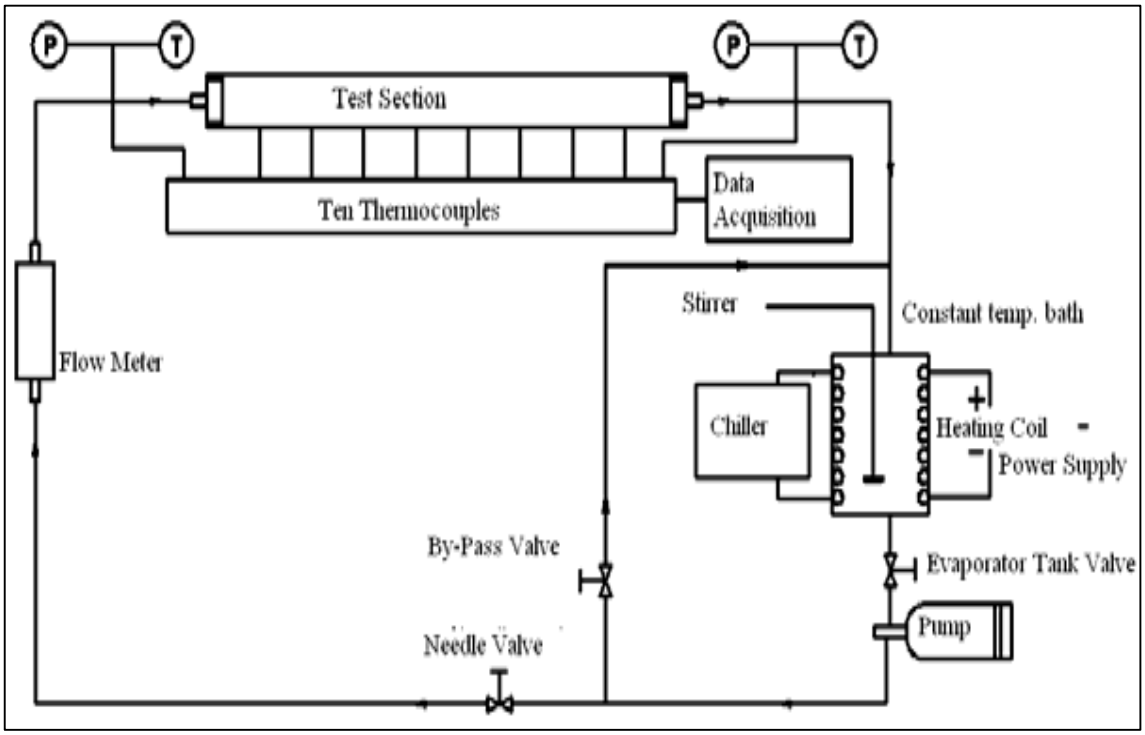

Figure 2:3 The experimental system of Asirvatham et al. (2009)

2.3 Literature on Turbulent Flow of Nanofluids Heat Transfer

Convective heat transfer enhancement is achieved by passive techniques either by changing the geometry of flow or by increasing the thermal conductivity of liquids. Since Maxwell‘s theoretical work on particle suspension[35], many theoretical and experimental works have been carried out on liquid suspensions containing solid particles of micro dimensions with the aim of increasing the convective heat transfer coefficient. The use of course grained particles was abandoned because of the instability of suspensions and possible erosion of pipe material by course grained particles.

When compared to micro sized particles, the nanoparticles provide relatively larger surface area and facilitate formation of stable suspensions and the problems of clogging, sedimentation and erosion are minimized to a greater extent. The use of nanofluids as working fluids in heat exchangers leads to miniaturization of its components. The progress in nanofluid development is hindered even today because of a lack of agreement among various research findings, poor characterization of nanofluids, and lack of a theoretical base to understand the mechanism of particle transport. The presence of nanoparticles in a base liquid changes its heat transfer performance considerably. For the commercial application of nanomaterials, appropriate theory needs to be developed to understand the mechanism of how the nanofluids exhibit higher and more favorable thermophysical properties.

Pak and Cho in 1998 conducted experiments to investigate the hydrodynamic and convective heat transfer behaviors of submicron particles of metal oxide in a turbulent flow regime with a constant heat flux condition. He, Jin et al. in 2007 investigated the

effects of particle size and concentration, and the flow rate of aqueous TiO_2 nanofluids in a vertical pipe considering the laminar and turbulent flow regimes.

Li, Xuan et al. in 2003 investigated the heat transfer behavior of copper nanoparticles suspended in water. In their experimental study, different particle volume fractions (0.3, 0.5, 0.8, 1, 1.2, 1.5 and 2%) were used to study the effect of nanoparticles on the heat transfer coefficient, with varying values of Reynolds number from 10,000 to 25,000. Their results exhibited an enhanced heat transfer process for suspended nanoparticles and also under the same Reynolds number; the nanofluids have a higher heat transfer coefficient than that of the base fluid. The Nusselt number of 2% volume fraction of Cu particle nanofluid was 39 % higher than water. Figure 2.4 shows the experimental system of their work.

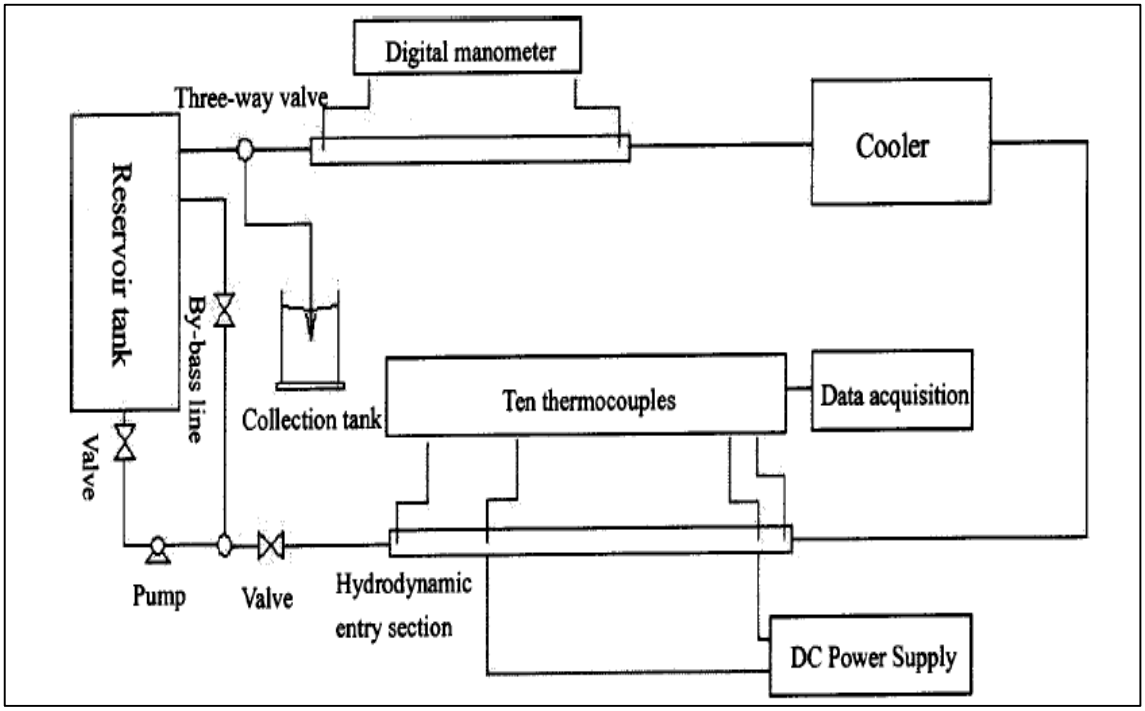

Figure 2:4 Experimental system of Xuan and Li

Nguyen, Roy et al. in 2007 studied the heat transfer behavior of water based Al_2O_3 nanofluids for the cooling of microprocessors. Their experimental results clearly demonstrated a 40% enhancement in the convective heat transfer coefficient for a nanofluid with 6.8% volume concentration. The increase of nanoparticle concentration in the base liquid resulted in the decrease of temperature in the heated block. Furthermore, nanofluids with 36nm particle size showed more heat transfer compared to the 47 nm particle size. Chen, Yang et al. in 2008 have studied synthesis, characterization and dispersion characteristics of titanate nanotubes in water. In their experimental work they investigated the thermophysical properties such as the thermal conductivity, the viscosity and the forced convective heat transfer of the nanofluids.

Li and Kleinstreuer in 2008 investigated the thermal performance of 1-4% volume concentration CuO-water nanofluids flowing in a trapezoidal micro-channel. The results clearly indicate that the thermal performance of nanofluids increases with an increase in the nanofluid volume fraction. However, nanofluids with higher particle concentration have shown a considerable increase in the pressure drop requiring more pumping power. They concluded that micro-channel heat sinks with nanofluids are considered to be promising as cooling devices. Koa in 2007 measured viscosity and pressure drop characteristics of nanofluids with carbon nanotubes present in the water and found that preparation methods of nanofluids and the concentration of carbon nanotube have a bearing on the pressure drop. Stable suspensions of nanotubes were prepared by two methods. In the first method a surfactant was used and in the second method acid was mixed in the nanofluids. Both the nanofluids have shown an increase in the nanofluid viscosity with a decreasing shear rate and pressure drop. Both the nanofluids presented

similar results when compared to base liquids because of the shear thinning nature of CNT nanofluids. It was also observed from the study that CNT nanofluids produced low friction factors compared to the conventional fluids alone, especially in the turbulent flow regimes.

Namburu, Kulkarni et al. in 2007 carried out a numerical study of the heat transfer characteristics of CuO, Al_2O_3 and SiO_2 nanoparticles in a base fluid of water and ethylene glycol under turbulent flow condition and blended applying constant heat flux boundary conditions. The results indicate that at a 20,000 Reynolds number the heat transfer coefficient of 6% CuO nanofluids increased by 175% over the base fluids. It is further noted that the heat transfer coefficient is a strong function of nanofluid volume concentration and Reynolds number. Employing nanofluids at higher temperature gives higher percentage increases in the heat transfer enhancement. The results also show that CuO nanofluids exhibited higher heat transfer enhancement followed by Al_2O_3 and SiO_2 nanofluids.

A recent study conducted by MinSheng Liu et al. in 2011, investigated the enhancement of thermal conductivity of nanofluids prepared by using a two-step method for dispersing copper oxide (CuO) and carbon nanotubes in water, ethylene glycol and synthetic oil without using surfactants. However, the one-step method was used for dispersing copper (Cu) in water, ethylene glycol and oil.

A modified transient hot wire method was used to measure the thermal conductivities of the nanofluids. Their results show that the nanofluids contain low volume concentration of used nanoparticles have greater thermal conductivity than that in the original fluids.

The experiment was carried out in a performance system of a 10-RT water chiller (air conditioner) as shown in Figure 2.5.

The test system flow rate ranged from 60 to 140 L/min. It was observed an about 4.2% increase in cooling capacity of nanofluids at a flow rate of 100 L/min. It was also shown that the heat performance coefficient of the water chiller increased by 5.15% relative to that without nanofluids. The researchers also concluded that the dynamic effect (dispersion) may effectively enhance system performance.

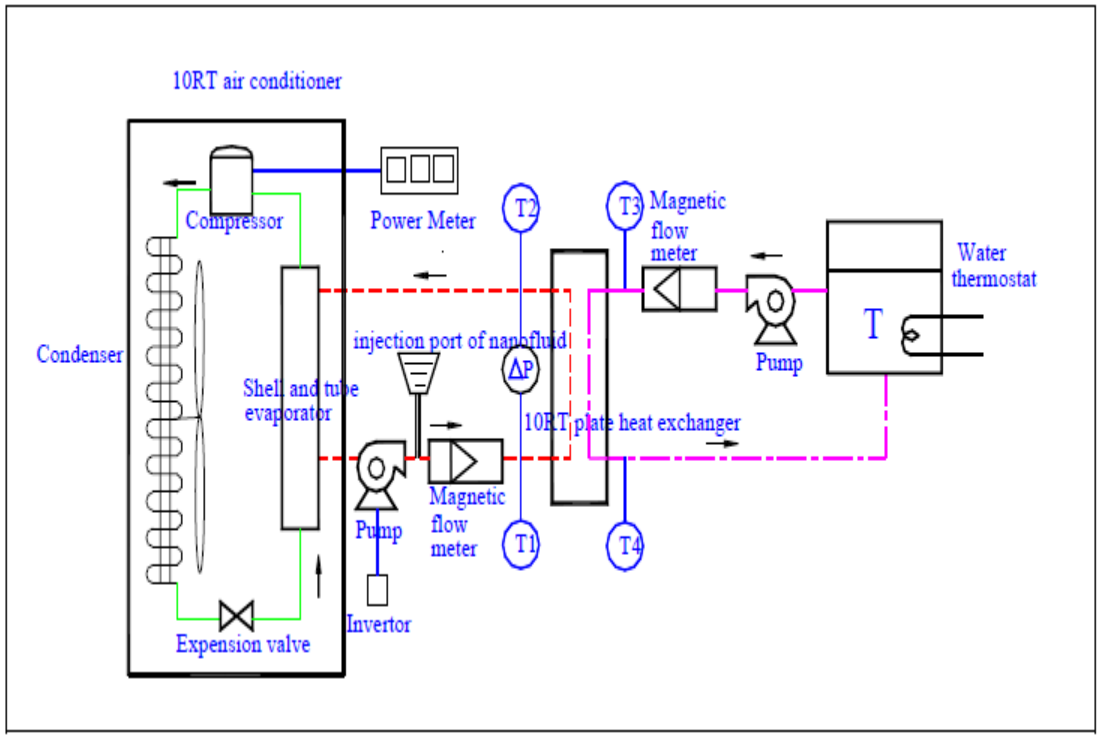

Figure 2:5 Experimental test system for (MinSheng Liu 2011)

2.4 Preparation of Nanofluids

The idea behind the preparation of nanofluids is to use them as thermo fluids in heat exchangers for improvement of heat transfer characteristics and thus to minimize the size of heat transfer equipment. Nanofluids help in conserving heat energy and heat exchanger material. The important considerations which affect the heat transfer features of nanofluids are the thermophysical properties such as thermal conductivity, specific heat capacity and viscosity. The thermophysical properties of nanofluids also largely depend on operating temperature of nanofluids. Hence, the accurate measurement of temperature dependent properties of nanofluids is essential.

Nanofluid preparation is an important process for dispersion of nanoparticles into base fluids. In 1993 Masuda found instability in fluids since the particles tend to agglomerate, while Grimm in 1993 conducted his experiments quickly because of particle settling. Nowadays, nanofluid preparation has been further developed; therefore, nanoparticles have high stability and dispersability in base fluids, as well as chemical compatibility with the host fluid. In general the main techniques that have been innovated to prepare nanofluids are the single-step and the two-step methods.

2.4.1 The Single-Step Method

In this method nanoparticles are produced and dispersed in the base fluid to form nanofluids during one cycle process. Eastman, Choi et al. in 2001 developed this technique by using a physical vapor condensation method to produce Cu-ethylene glycol nanofluids. This process includes simultaneously producing and dispersing the nanoparticles in the fluid. Many processes are avoided such as drying, storage, transportation, and dispersion of nanoparticles, so this will minimize the nanoparticle agglomeration and hence, the stability of fluids is improved.[51]

Comparatively to the two-step technique, the nanofluids that are obtained by direct evaporation present less agglomeration issues [47],[45]. Amongst the technique limitations, the most significant is the small sample size, which limits the process mass production for commercialization [47],[48]. Another limitation is the low vapor pressure restriction for liquids. Some of which can vaporize at low to moderate temperatures [47]. Particle size control is also an issue when employing this technique [45], [47].

Size control need not be a problem as it is possible with the chemical vapor condensation technique. Nanoparticle powder is directly deposited into the base fluids, which results in higher clustering than direct evaporation. Chemical synthesis techniques, such as precipitation, can produce small, mono-dispersed nanoparticles with reduced agglomeration. However, the effect of surface molecules on the nanofluid thermal properties remains unknown. Few studies have employed nanofluids obtained using such techniques. CNTs have also been obtained via the single-step technique such as carbon plasma discharge directly in water. [47],[49].

2.4.2 The Two-Step Method

This method is widely used for preparing nanofluids. This process firstly produces dry powders such as nanoparticles, nanotubes, and nanofibers by using chemical or physical methods. Secondly these nanosized powders are dispersed into the base fluid with the help of ultrasonic agitation, magnetic force agitation, high-shear mixing, ball milling and homogenizing. This method is the most economic, large-scale process for producing nanofluids, because these synthesis techniques for nanopowders have already been scaled up to industry level. The enhancement of the nanoparticle stability is the most important step for the preparation of nanofluids. This can be achieved by using surfactants since the nanoparticles have high surface area and surface activity, and tend to aggregate. Figure 2.6 shows two types of nanofluids with and without surfactant.

Nan et al. in 2004 used a two-step technique for a carbon nanotube suspension. They also used surfactants and applied ultrasonic agitation to prevent CNT agglomeration. Hong, Yang et al. in 2005 and Hong et al. in 2006 dispersed Fe nanopowder in ethylene glycol using a two-step method. A chemical vapor condensation technique was used to synthesize iron. They applied an ultrasonic cell disrupter to prevent the nanoparticle agglomeration in nanofluids. Murshed, Leong et al. in 2005prepared nanofluids by dispersed TiO_2in water using the two-step method.

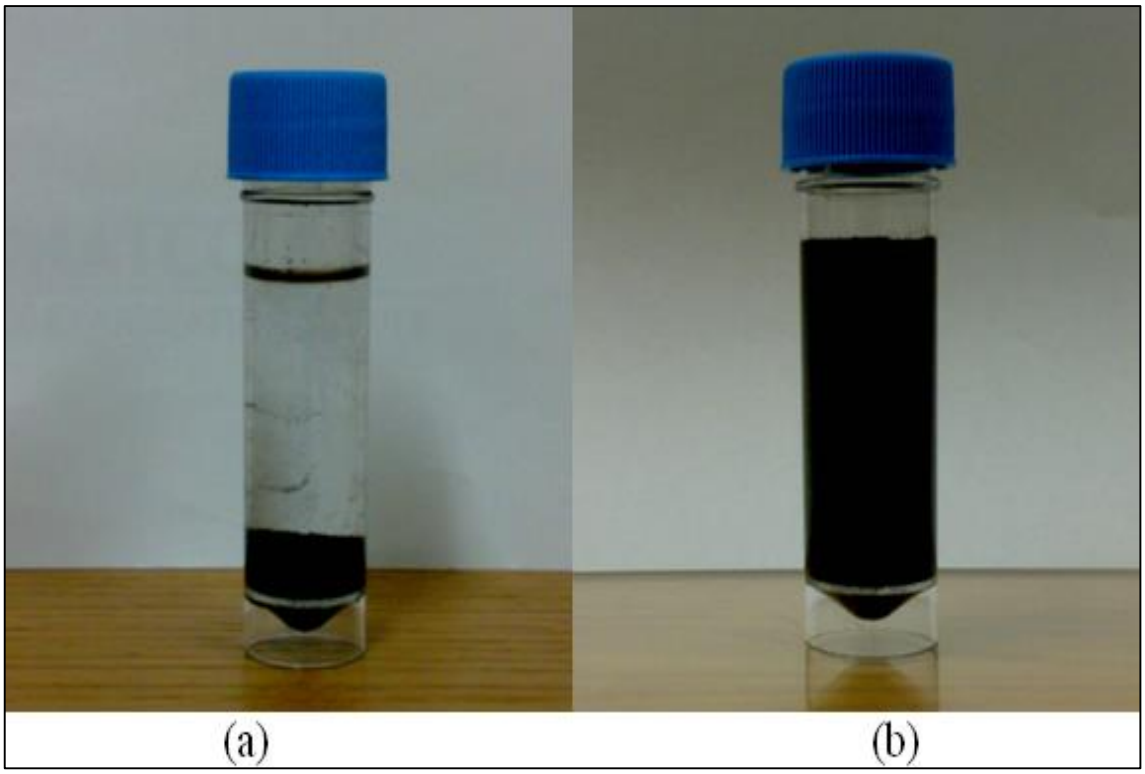

Figure 2:6 In (a) without surfactant the CNTs rapidly settle down at the bottom, whereas in (b) CNTs are well dispersed in water with surfactant.

CHAPTER THREE

3 EXPERIMENTAL METHODOLOGY

This chapter discusses the principles and methods of different experimental techniques used to prepare the nanomaterials and the nanofluid and characterize them. Different techniques were used to characterize the nanomaterials such as Scanning Electron Microscopy (FE-SEM), Transmission Electron Microscopy (HRTEM), Thermo-Gravimetric Analysis (TGA) and Fourier Transform Infrared Spectroscopy (FTIR)**.**

The characteristic thermal properties such as thermal conductivity and specific heat capacity of the prepared nanofluid samples were measured by a set of thermal techniques such as Differential Scanning Calorimetry (DSC) and Thermo-Gravimetric Analysis (TGA). The other property of the nanofluids such as viscosity was measured using the Stormer viscometer.

3.1 Materials

3.1.1 Raw Multi-Walled Carbon Nanotubes (MWCNTs)

Multi-walled carbon nanotubes (MWCNTs), purchased from Chengdu Organic Chemicals Co. Ltd. "TimesNano", China with the following specifications: 10 to 20 nm outer diameter with length from 10 to 30 μm, 200 m^2/g specific surface areas and 95% MWCNT purity, were used in this study.

3.1.2 Impregnation of Carbon Nanotubes with Metal Nanoparticles

The iron oxide (Fe_2O_3), Aluminum oxide (Al_2O_3) and copper oxide (CuO) nanoparticle doped on CNTs were prepared by an impregnation method. The precursors used were iron nitrate, aluminum nitrate and cooper nitrate from Sigma and Aldrich, with 99.9% purity. The metal salt was first dissolved in alcohol at different concentrations in order to impregnate the surface of CNTs with various percentage loads. After dissolving the salt in alcohol, fixed amount of CNTs were added to the aqueous solution and sonicated for 30 minutes using probe sonicator. The aim of the ultrasonication process was reduce the agglomeration of the CNTs and increase their dispersion in order to have maximum contact between the CNTs and metal salt. Then the sample was dried in vacuum oven and calcinated at 350 ^{o}C for 3 hr.

3.1.3 Functionalized CNTs

The functionalized CNTs were prepared by oxidizing the raw CNTs by nitric acid. The oxidation process, illustrated in Figure 3.1, usually removes the metallic catalysis from CNTs by dissolution. This procedure minimizes the shortening of the tubes and the chemical modification is limited mainly to the tube caps and to the side walls of the defected sites.

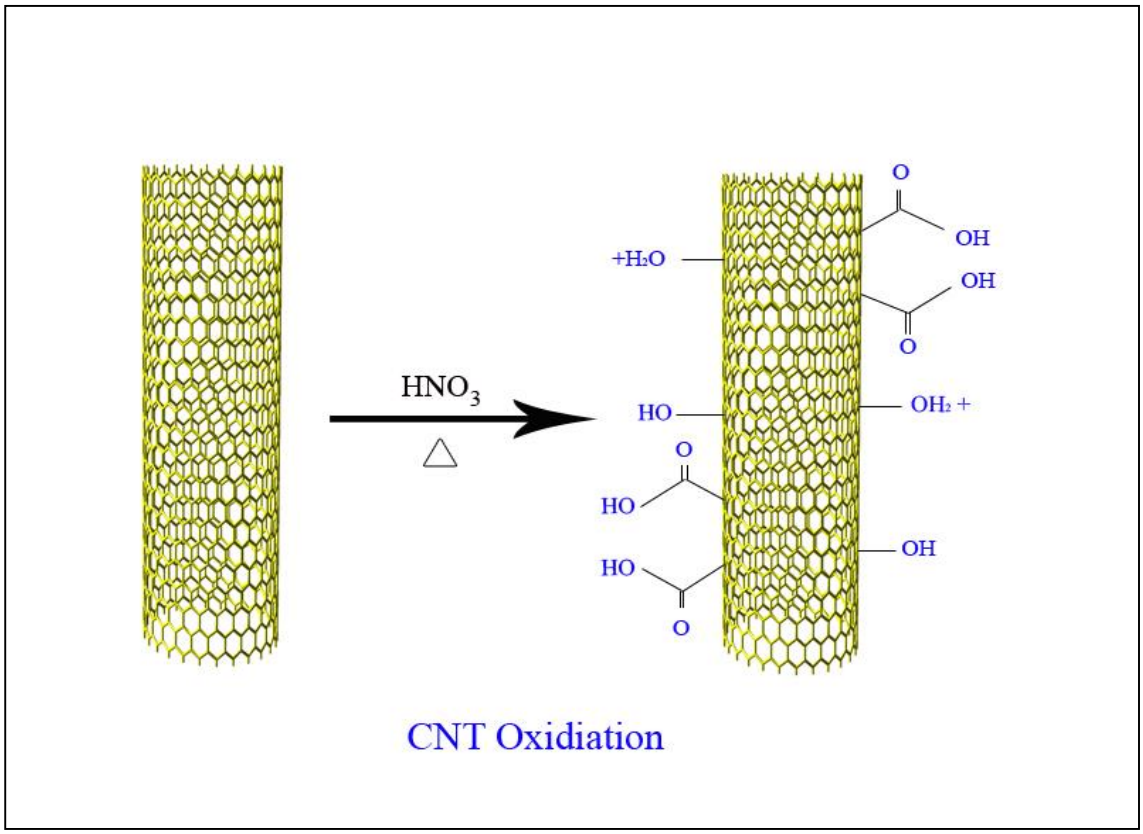

Figure 3:1: Chemical Functionalization of MWCNTs through the use of chemical thermal oxidation

3.2 Functionalization of CNT with polyethylene glycol

A portion of the oxide/washed carbon nanotubes (COOH-CNT) was functionalized with polyethylene glycol. The oxidized CNT has active sites enabling to form chemically attached bounds with the surface of the CNT. The experimental procedure started by mixing an excess amount of polyethylene glycol with certain amount of oxidized CNT in a 500 ml beaker, and then melting the mixture at the melting point of PEG (~80°C) using a hot plate while stirring. The amount of melting PEG should be enough to allow submerging all used CNT. After that, a few drops of acids like sulfuric acid or nitric acid were added to the mixture as a catalyst. The reaction was kept on a hotplate while stirring for 2 to 3 hours. The reaction mechanism is illustrated in figure 3.2. After achievement of the reaction, the mixture was dissolved in an excess amount of toluene several times and then filtered by vacuum through a filter paper; this adding toluene process was used to remove any traces of un-reacted PEG and sulfuric acid from the produced CNT-PEG. Finally, a draying process was applied at a temperature of 80^{o}C using a vacuum oven to evaporate toluene from the produced powder of the CNT-PEG.

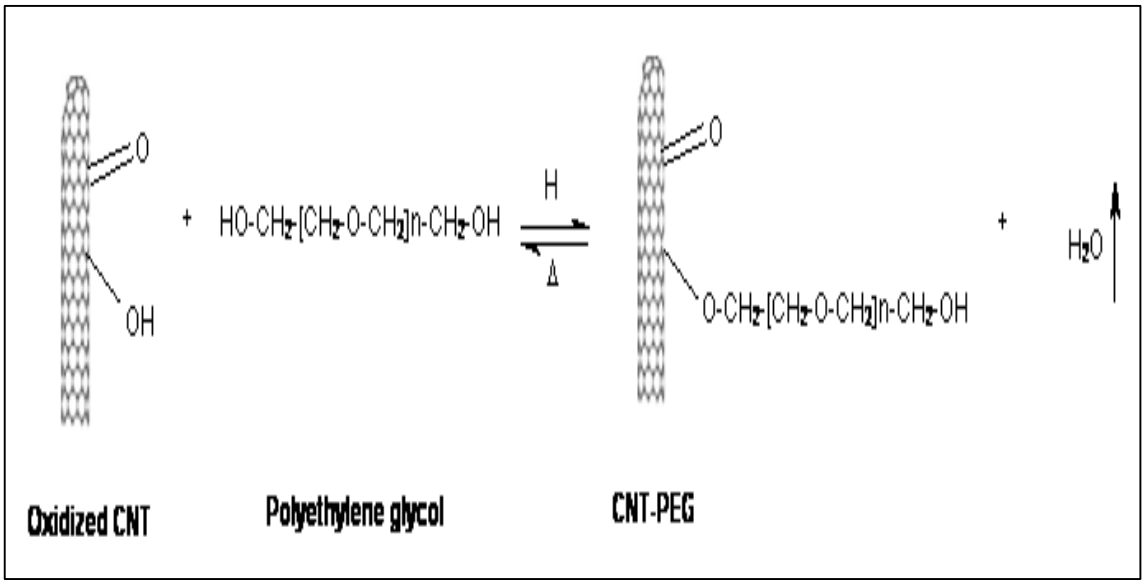

Figure 3:2 : The chemical Reaction of Oxidized CNT with PEG

3.3 Impregnation of Metal Nanoparticles on CNTs by Wet Impregnation Technique

The iron-, copper- and aluminum-oxide nanoparticles were prepared by impregnation method. Different weight loading of the selected metal salts were dissolved in ethanol solution in an appropriate concentration to obtain desired metal loading on the surface of the carbon materials within the range 1-10 wt%. The impregnation was performed under ultrasonic condition for 30 minutes at a fixed 25^{o}C temperature controlled by the water bath. Every batch was prepared from an amount of 0.5gram of carbon in a 32.7ml of glass bottle sample with 3cm in diameter. Ultrasonification instrument were used in this preparation. The aim of ultrasonication during impregnation is to have a complete and homogeneous wetting of the whole sample particles during impregnation. For the drying step, all samples were dried in a conventional oven for 12 hours at 60℃ as shown in figure 3.3. Finally the samples were subjected to calcination step at 350 ℃ for 6 hr to produce iron oxide nanoparticles on the surfaces of carbon materials.

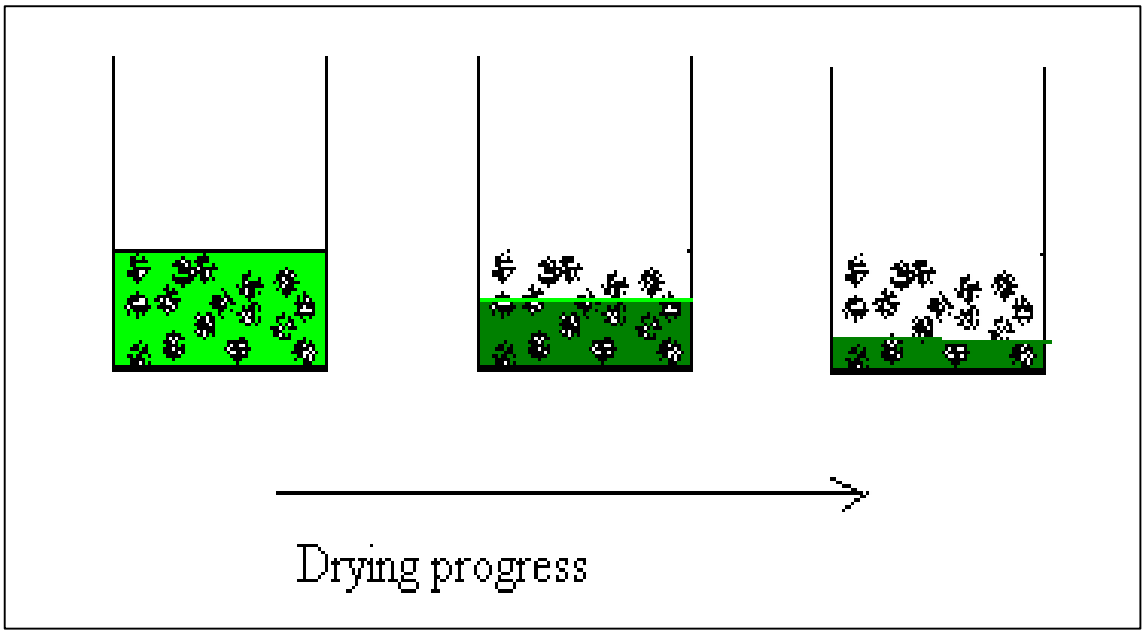

Figure 3:3 The increasing concentration of the metal salt solution during drying

3.4 Preparation of Nanofluids

The two-step method, which is a process by dispersing nanoparticles into base liquids, was used for preparing nanofluids. Deionized water with functionalized and impregnated CNT was used to produce nanofluids**.** In case of functionalized CNT, nanofluid was prepared by adding the calculated amount of functionalized and impregnated CNT in a known amount of deionized water base fluid in a beaker and sonicated for 30 min. The ultrasonic pulses of 750 W and 20 kHz generated by an ultrasonic cell disrupter (shown in Figure 3.4) was used to improve the dispersion of CNT in the base fluid.

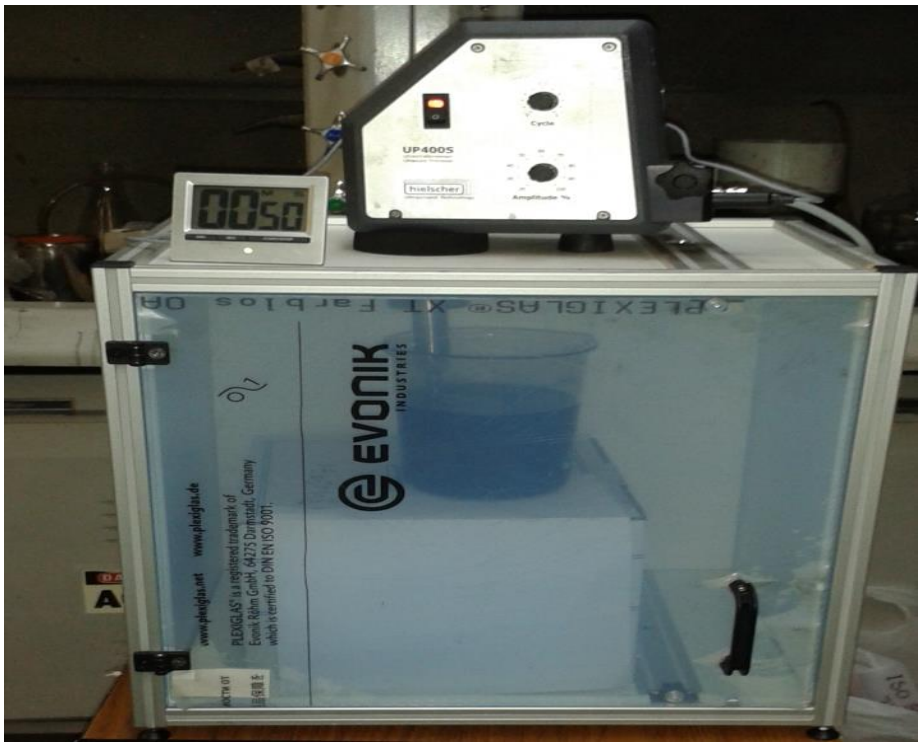

Figure 3:4 Photograph of an ultrasonic cell disrupter

3.5 Characterization

The functionalized and impregnated CNTs were analyzed using Fourier Transform Infrared Spectroscopy (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM) and Energy Dispersive X-ray analysis (EDX).

3.5.1 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) has shown limited ability to probe the structure of MWCNTs. A factor that has hindered the advancement of FTIR as a tool for MWCNT analysis is the poor infrared transmittance of MWCNTs. A solution to this is the use of KBr preparations of nanotube samples. Because of their black character and strong absorbance, MWCNT are often unable to be distinguished from background noise making it necessary to use a very weak concentration of the nanotubes in a KBr powder. However the greater vibrational freedom of attached polymeric species presents much more pronounced peaks and are typically the focus of attention in FTIR results. Despite this, with very careful sample preparation some researchers have managed to elucidate peaks corresponding to surface bound moieties such as carboxylic acid groups at wavenumbers of 1791, 1203 and 1080cm^{-1}. The spectra of our samples were recorded by PERKIN ELMER 16F PC FT-IR instrument. FTIR samples were prepared by grinding dry material into potassium bromide adding of ~0.03 %wt. This very low concentration of MWCNTs was necessary due to the high absorption of the nanotubes.

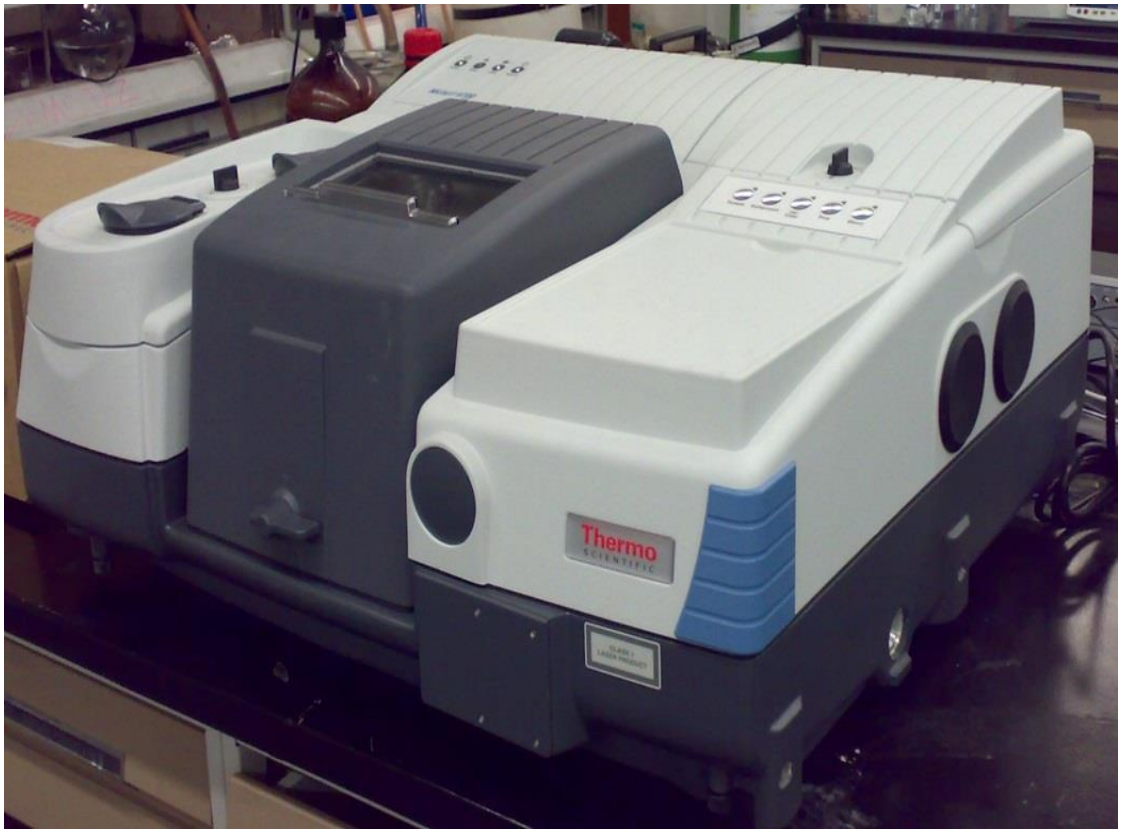

Figure 3:5 : Photograph of Nicolet 6700 FT-IR Spectrometer.

3.5.2 Differential Scanning Calorimetry (DSC)

Differential scanning Calorimetry is an instrumental technique for the measurement of changes in enthalpy as a function of temperature. In this technique, the sample of material is generally exposed to a linear temperature, so the rate of heat flow is continuously measured for the sample; this is proportional to the instantaneous specific heat.

In this work, differential scanning calorimetry, (DSC Q1000, Model TA Instruments, USA), connected with rapid cooling system, RCS 90 using Nitrogen gas, was used.

In order to measure the specific heat capacity (C*p*) of nanofluids, the samples were sonicated for 10 minutes and then placed in the DSC. The DSC curve is the plot of the specific heat capacity as function of temperature. The thermal properties of the nanofluid samples investigated in this work were analyzed by measuring nanofluids sample weights (about 20 mg) in hermetic aluminum pans and lids. The heat range was applied varying from 25^{o}C to 50^{o}C with minutes and cooled in an ice bath to room temperature immediately before being a 1.5^{o}C/min heating rate.

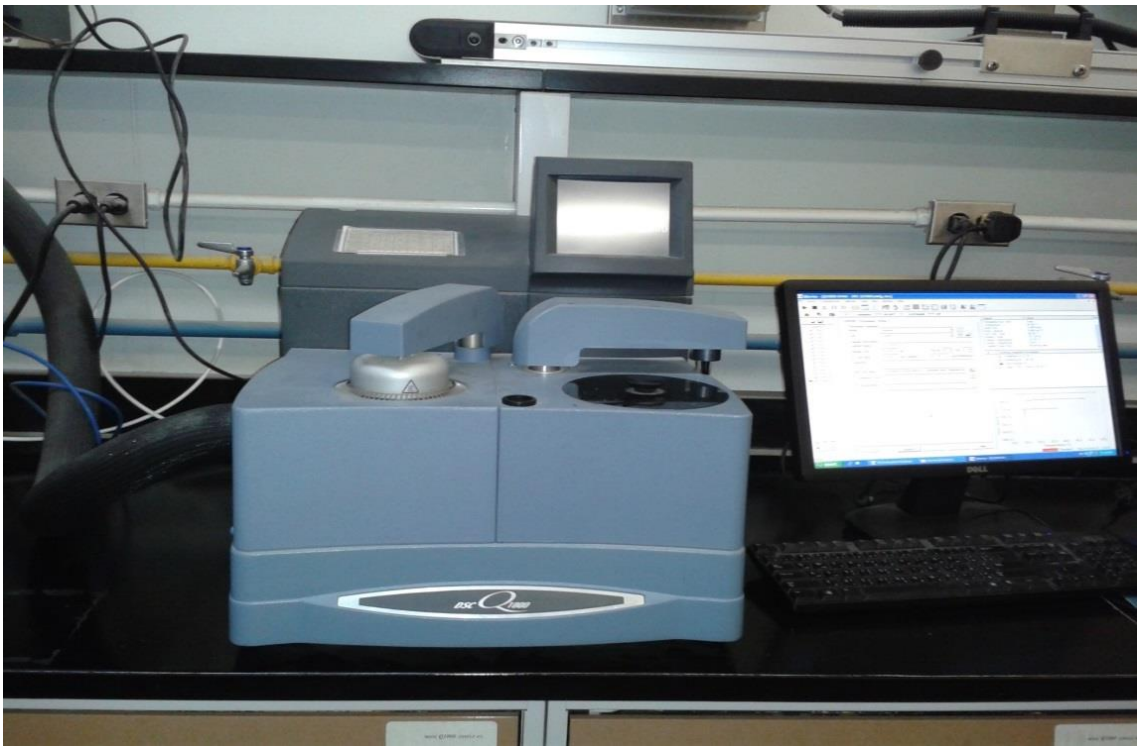

Figure 3:6: Photograph of DSC Q1000, Model TA Instruments.

3.5.3 Thermo Gravimetric Analysis:

Thermo gravimetric analysis (TGA) is a thermal analytical tool used for determining a thermal stability and the volatile components through monitoring the changing in specimen weight that occurs during heating. The measurement is normally carried using argon as inert gas, and recording the weight as a function of temperature. The TGA can give information such as composition of multi-component systems, oxidative stability, and thermal stability, estimated lifetime of a product, decomposition kinetics, moisture and the volatile component of a material. These components were included in a container like crucible to hold the sample, a furnace to heat the sample to a high temperature, and a suitable balance for continuously monitoring the sample weight. The tested material was placed in an aluminum cup supported on an analytical balance located outside the furnace chamber.

The TGA plots a curve of the change in the weight percent on the Y-axis versus the reference material temperature on the X-axis.

A Netzsch model STA 449 F3 Jupiter® TGA was used in this study gravimetric analysis. The TGA was used in this study not only to detect the moisture but also the amount of surfactant present. The measurements of weight loss were taken from 25 to 1400 ^{o}C with 20^{o}C/min increment temperatures. Argon was used as the inert gas.

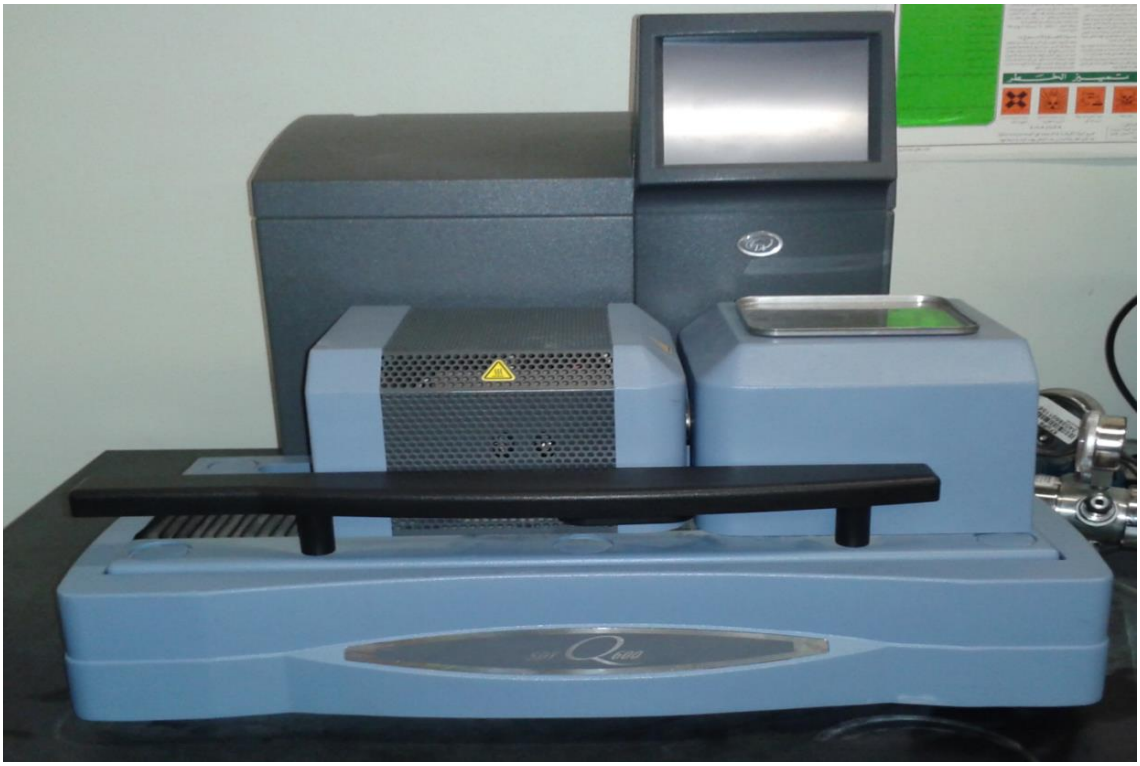

Figure 3:7 : Photograph of Netzsch model STA 449 F3 Jupiter® device.

3.5.4 Scanning Electron Microscopy (SEM)

Scanning electron microscopy is a widely used technique to visualize and analyze the surface morphology and micro structural characteristics of materials.

A scanning electron microscope (SEM) generates a range of signals to the surface of specimens through uses a focused beam of high energy electrons. The signals which are resulted from electron sample interactions reveal the sample information such as surface morphology, crystalline structure and chemical composition.

In most of the applications the data are collected over a selected area of the surface of the sample, and a 2D image is generated that displays spatial variations in the surface properties of a material. In SEM, a sample is exposed by an electron beam and data on the sample are transported by secondary electrons emitted from the surface layer of thickness ~ 5nm and by backscattered electrons released from the volume of linear size ~ 0.5μm.

In this work scanning electron microscope (MIRA3 FEG-SEM) having an image resolution of ~ 3nm at an accelerating electron voltage of 15kV**,** equipped with energy dispersive X-ray (OXFORD Instruments X-MAX) as shown in Figure 3.7 was used. It was used to observe the morphology and the elemental composition of impregnated carbon nanotubes with aluminum oxide, copper oxide and iron oxide.

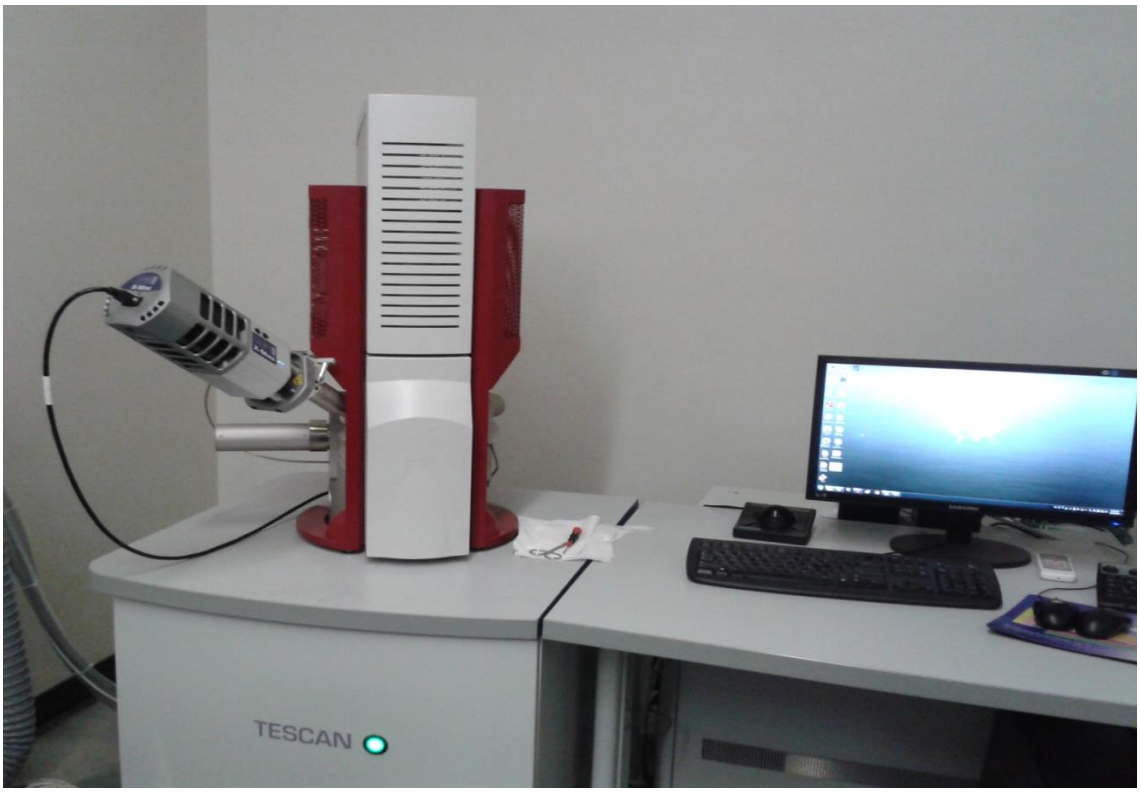

Figure 3:8: **Photograph of Scanning Electron Microscope (MIRA3 FEG-SEM).**

3.5.5 Energy Dispersive X-ray Spectroscopy (EDX)

Energy dispersive X-ray spectrometer (EDX) attached to the scanning electron microscope analyzes characteristic X-ray radiation emitted from the specimen when the electron beam interacts with the specimen. The main use of EDX is to accurately determine the composition of the sample under investigation. Upon exposing the samples to high energy electron beams the various atoms present in the sample emit characteristic X-rays which can be observed as several distinct peaks on an energy scale. The intensities of the peaks can be compared with the peaks of a standard sample to obtain the relative amounts of each atomic species, whereby accurate composition of the sample can be determined.

3.5.6 Viscosity Measurement

There are various methods for experimental measurement of viscosity, namely, the concentric cylinder, cone and plate methods. The technique chosen in the present study was the Stormer viscometer. As shown in Figure 3.8, this device was used to measure the kinematic viscosity of the nanofluids. This viscometer is preferred, because the meniscus of the nanofluids is not clearly visible due to the opacity of the fluid. The kinematic viscosity was calculated through measuring the time needed for an internal cylinder to make 100 revolutions in response selected weight.

In the present study, the measurements were taken for temperatures ranging from 25^{o}C to 65^{o}C for different volume concentrations of nanofluids. In order to have the experimental values of viscosity at different temperatures, a hot-plate was placed under the fixed

cylinder. Also water was used as the heating fluid in the fixed cylinder, which was thermally insulated.

Then the measurements were taken and the same procedure was repeated for different temperatures and volume concentrations. The uncertainty in the value of the viscosity of nanofluid measured by this equipment is less than 1.5 % of its mean value for the entire range.

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Figure 3:9: Photograph of Stormer viscometer.

3.6 Experimental Setup and Calculations

3.6.1 Shell and Tube Heat Exchanger

The experimental set up constructed for this work is shown schematically in figure 3.10. The unit mainly consists of a concentric tube heat exchanger. The test section consisted of 3 m annular tube which was constructed of 10 mm diameter inner stainless steel tube with 1 mm thickness and 23.4 mm diameter outer glass tube. In order to minimize the heat loss, the hot fluid (nanofluid) flows in the inner tube, while cold in outer tube. Two water baths were provided to keep the fluid circuits at constant temperature. Four thermocouples (K-type) were used to measure the inlet and outlet temperatures. The nanofluids, with different particle weight percentages, were used to determine the effect of the nanoparticle concentration on effectiveness of heat exchanger; the percentages of the nanoparticles were varied from 0.01 -to- 0.1 wt% which was controlled by digital mass flow controllers. Experiments were performed for mass flow rates ranging from 200 kg/hr to 640 kg/s. The temperature of the inlet tube and shell were varied from 20-35 ^{0}C. Two digital pressure transducers were connected to the inner and outer tubes in order to measure pressure difference at low and high flow rates. The unit was monitored and controlled by a computer.

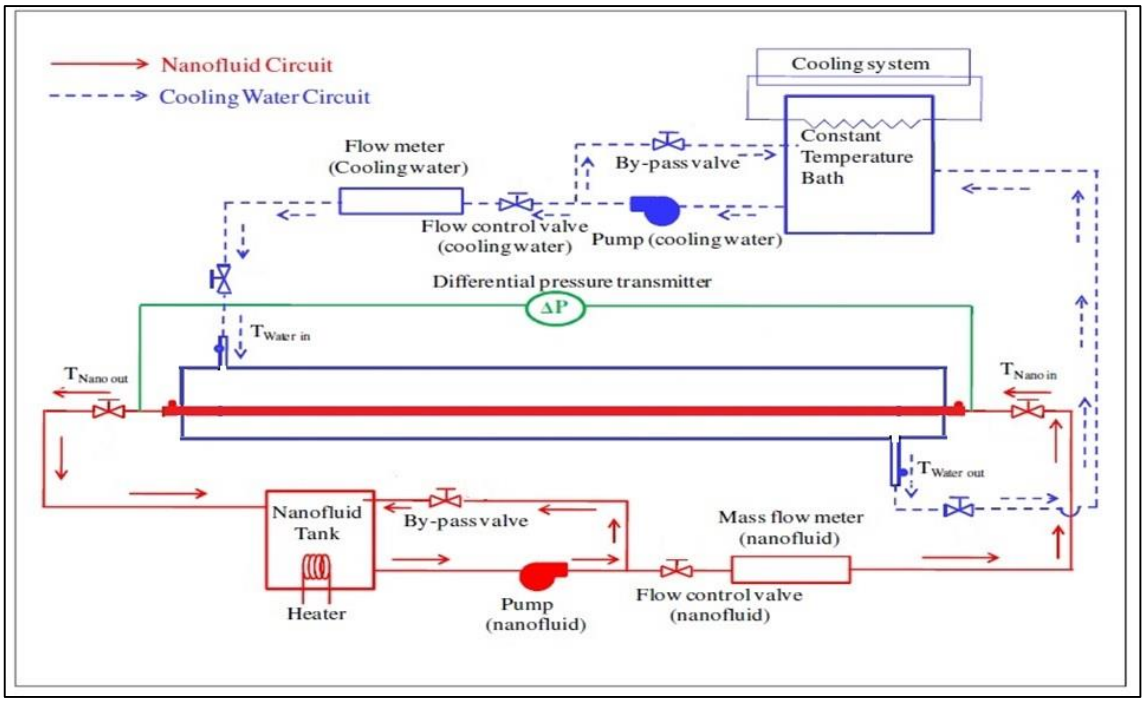

Figure 3:10: Shell and tube heat exchanger.

3.6.2 Nanofluid Heat Transfer Measurement

The fluid attained a steady state condition after about 30 minutes of turbulent flow. During experimental runs, the inlet and outlet temperatures of tube and shell and the pressure drop of tube side were measured at steady state conditions. From these data, the heat transfer enhancement was calculated using the following formula:

$$Q = \dot{m}_n \, Cp_n \left(T_{n,in} - T_{n,out} \right) \tag{3-1}$$

CHAPTER FOUR

4 RESULTS AND DISCUSSION

This chapter presents the results and discussions of the research finding. The results and discussions have been divided into four sections. The first part concerns the characterization of raw MWCNTs, impregnated MWCTS with metal oxide and functionalized MWCNTs which were investigated using scanning electron microscopy, Fourier transfer infrared (FTIR) and thermogravimetric analysis (TGA). Part two discusses the characterization of the temperature-dependent thermo-physical properties of nanofluids such as the specific heat capacity and viscosity of nanofluids using differential scanning calorimetry (DSC). The third part reports the experimental investigation of the convective heat transfer characteristics of nanofluids in a tube-in-tube counter flow heat exchanger and the pressure drop characteristics. The final part addresses the proposed mechanisms of nanofluids behavior in steel tube.

4.1 Surface characterization of raw and impregnated CNTs

This section discusses the characterization of unmodified carbon nanotubes and impregnated carbon nanotubes with iron oxide, copper oxide and aluminum oxide using scanning electron microscopy (SEM), back scattered FE-SEM, Transmission Electron Microscopy (TEM) and Energy Dispersive X-ray analysis (EDX).

Figure 4.1 (a & b) displays the FE-SEM images at low and high magnifications of raw carbon nanotubes. The diameters of the CNTs varied from 20 to 40 nm with an average

diameter of 24 nm, while the length of these carbon nanotubes ranged from 10 to 30 µm. This study was also supported by the characterization of doped CNTs with metal oxide (iron oxide, copper oxide and aluminum oxide) nanoparticles using back scattered FE-SEM in order to verify the presence of nanoparticles ions on the surfaces of the CNTs as shown in figures 4.2, 4.3 and 4.4.

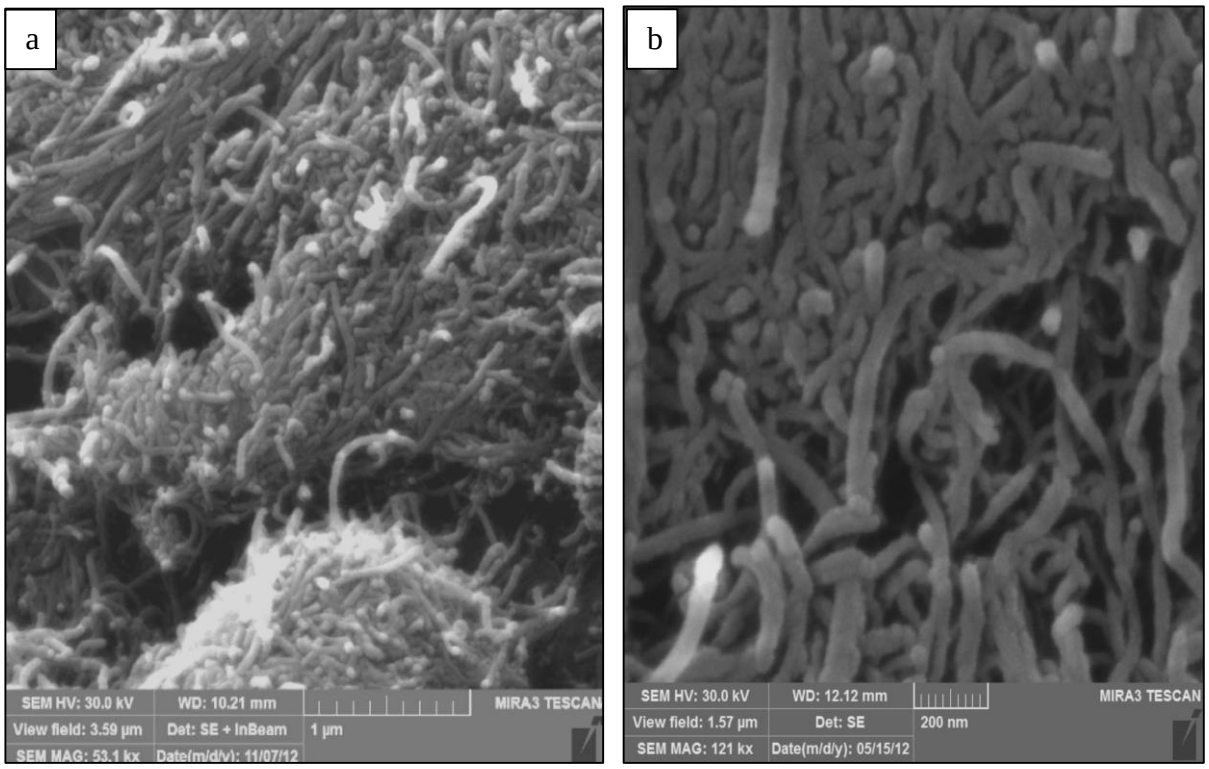

Figure 4:1 FE-SEM images of CNTs at (a) low magnification (b) high magnification

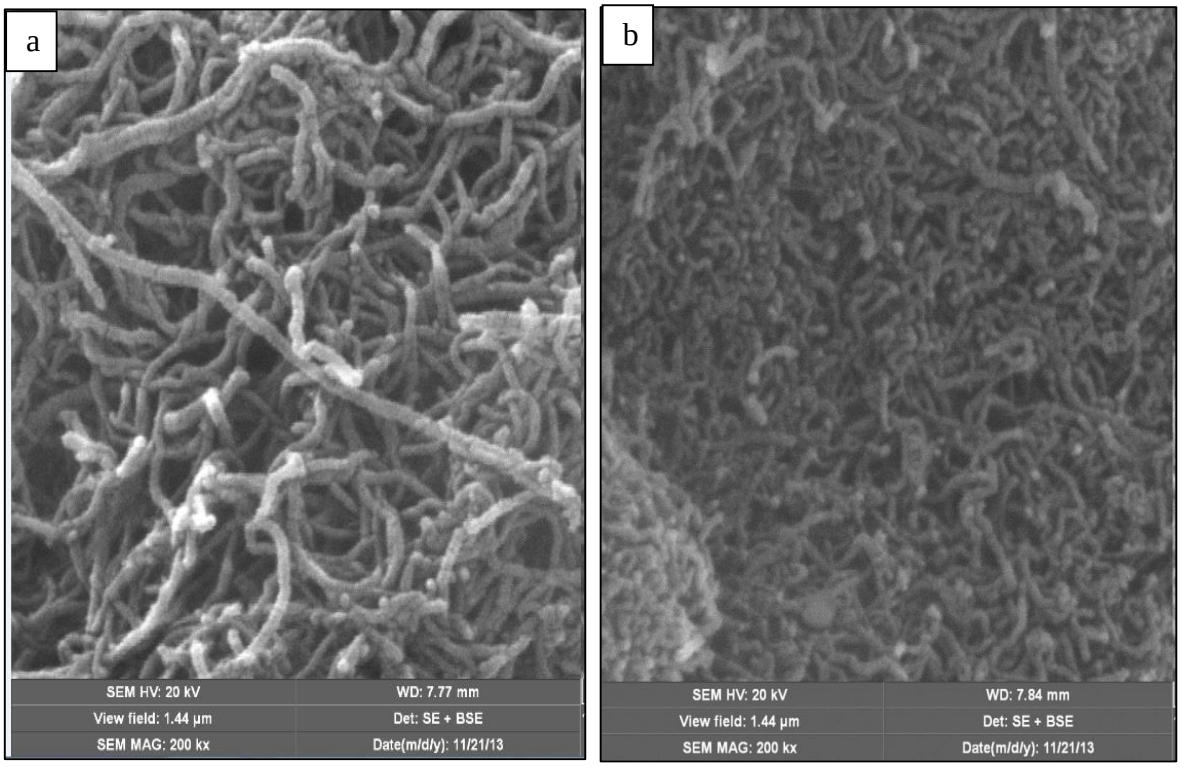

Figure 4:2 Back scattered FE-SEM images of CNTs doped with copper oxide nanoparticles with (a) 1 wt % and (b) 10 wt %.

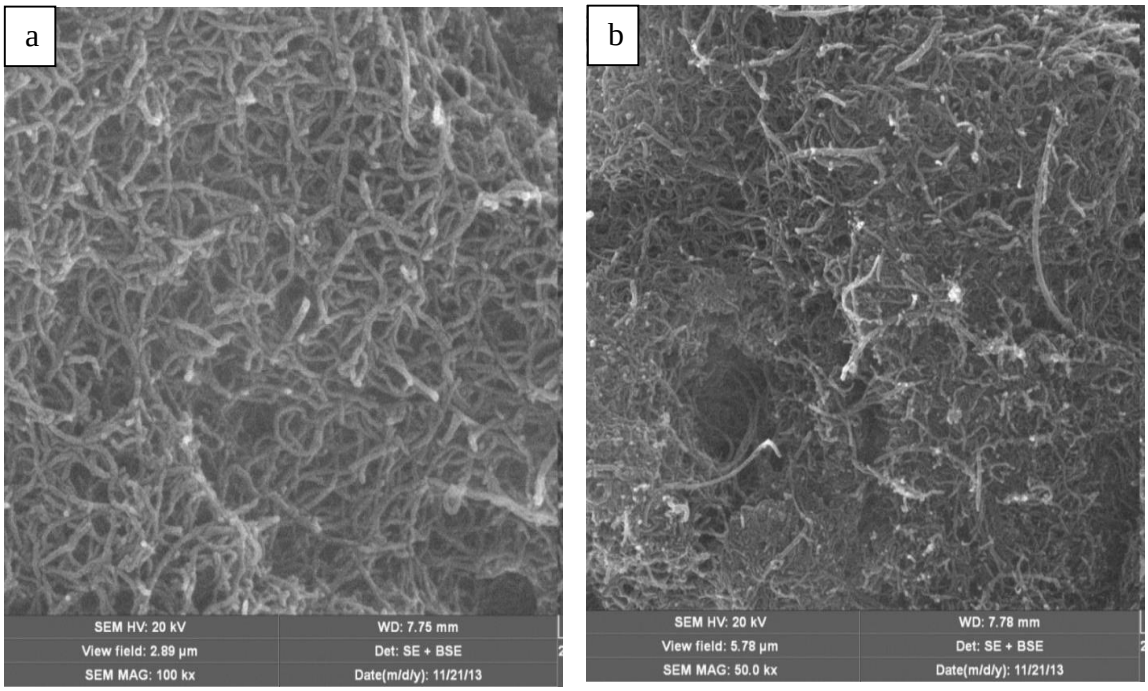

Figure 4:3 Back scattered FE-SEM images of CNTs doped with aluminum oxide nanoparticles with (a) 1 wt % and (b) 10 wt % .

The back scattering images were taken for two different samples at 1 and 10 wt. % of metal nanoparticles doped on CNTs as shown in figure 4.4. It was observed that, there are many formations of white crystal structures of metal nanoparticles with small sizes and irregular shapes. The distribution and agglomeration of metal oxide nanoparticles was also observed. At low concentration of metal nanoparticles (1 wt. %), the particles spread widely on the surfaces of carbon nanotubes forming very small crystals particles with diameters varying from 1-5 nm while at high concentration of metal oxide nanoparticles (10 w.t %) the particles were agglomerating forming a big crystals nanoparticles with diameters varying from 1-15 nm.

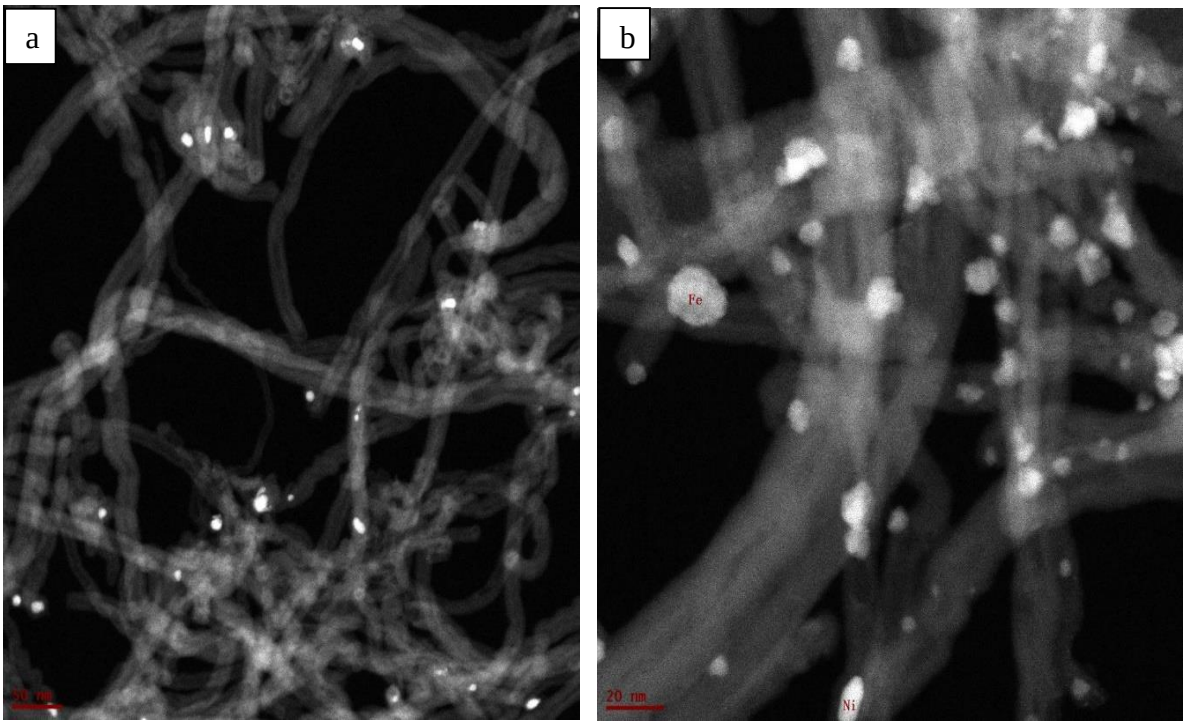

Figure 4:4 Back scattered FE-SEM images of CNTs doped with iron oxide nanoparticles with (a) 1 wt % and (b) 10 wt %

In order to identify the state of the metal oxide particles on the CNTs, elementary diffraction X-ray EDX analysis was conducted along with FE-SEM studies. The outcome from this analysis is shown in figures 4.5, 4.6 and 4.7 in which metals particles were confirmed to be in oxide forms. The peaks in figure 4.5 indicate the percentage of iron oxide nanoparticles coated on the surface of CNTs.

In figure 4.6 and figure 4.7, the strong peaks are assigned to copper and aluminum elements respectively, this indicates that the loading of copper and aluminum nanoparticles onto the MWCNT was performed successfully using wet impregnation method. It is clear that the peak on right hand side in each figure is higher than that on the left hand, this confirms that, almost 10 % of copper /aluminum oxide nanoparticles were doped on the surface of CNTs.

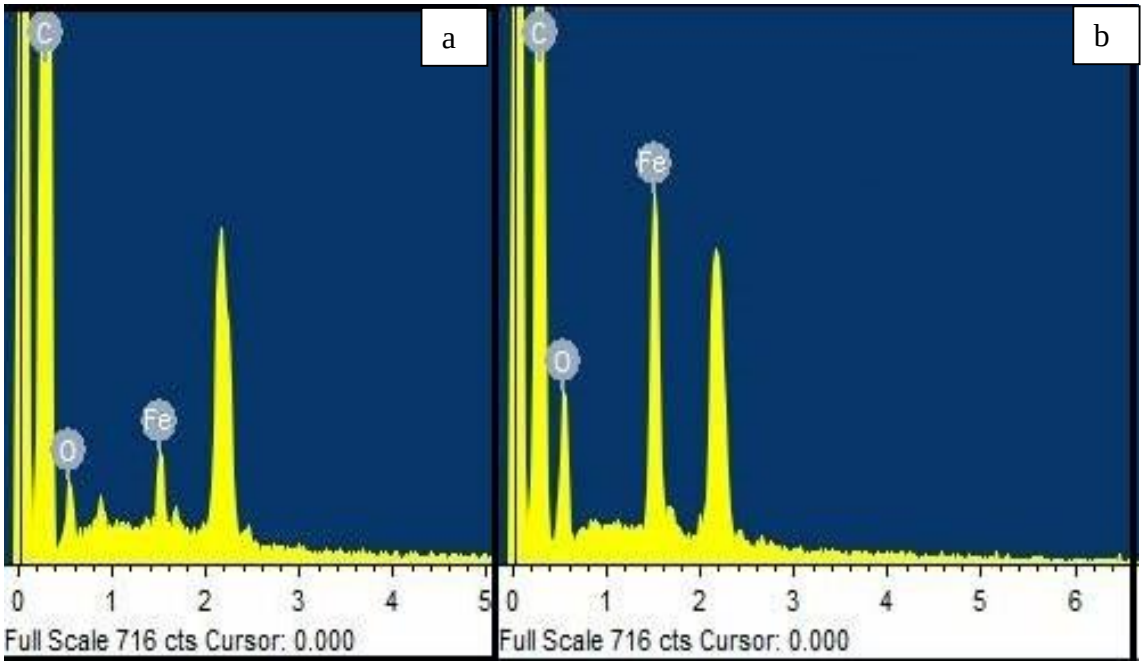

Figure 4:5 EDS analysis of CNTs doped with iron oxide nanoparticles (a) 1 wt % and (b) 10wt %.

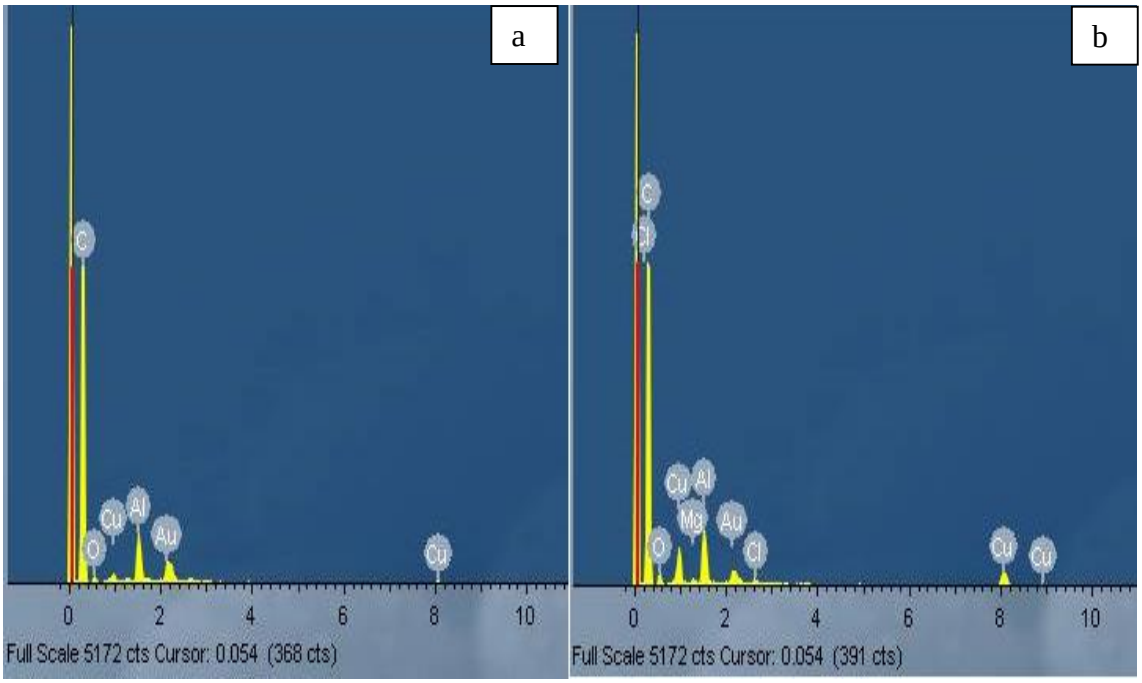

Figure 4:6 EDS analysis of CNTs doped with copper oxide nanoparticles (a) 1 wt % and (b) 10 wt %

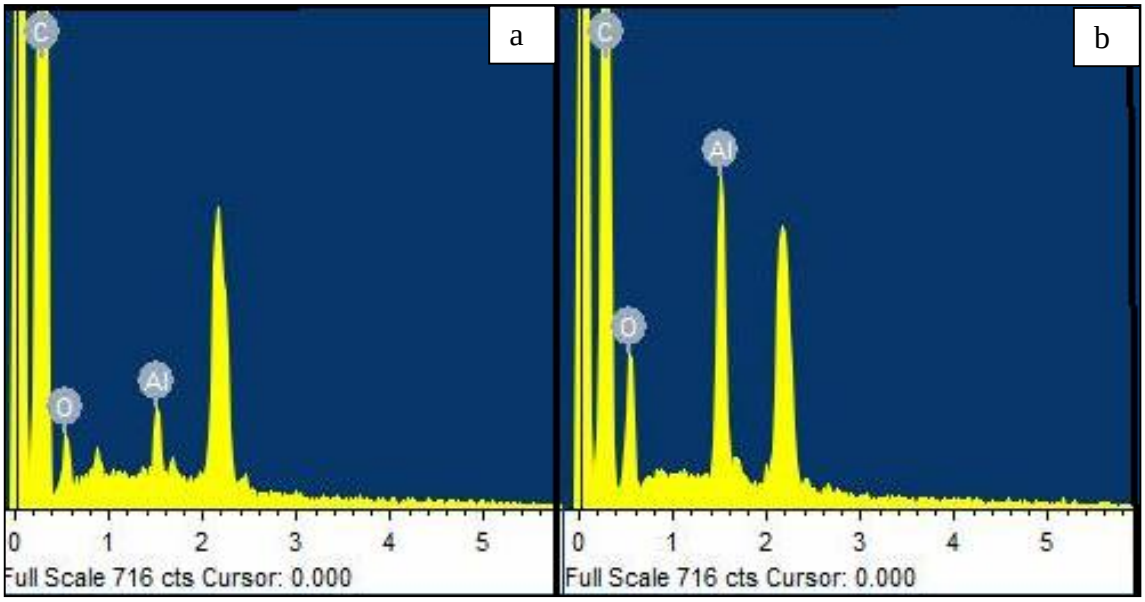

Figure 4:7 EDS analysis of CNTs doped with aluminum oxide nanoparticles (a) 1 wt % and (b) 10 wt % .

TEM was carried out to characterize the structure and size of CNTs and iron nanoparticles, and to observe the doping of iron nanoparticles onto the surface of CNTs. To prepare TEM samples, some alcohol was dropped on the nanotubes film, then, these films were transferred with a pair of tweezers to a carbon-coated copper grid. A representative TEM image of CNTs is presented in figure 4.8 (a). It is obvious, from the images that all CNTs are hollow and tubular in shape with many deflections sites. In some of the images, catalyst particles can be seen inside the nanotubes. Figure 4.8 (b) shows a TEM image of typical decorated CNTs. It was found that the iron nanoparticles cover the surfaces the CNTs with an average particle size of ~ 6 nm which is consistent with back scattered FE-SEM images. It was also noticed that, the structure of CNTs is not destroyed during the impregnation of nanoparticles on the surfaces of CNTs.

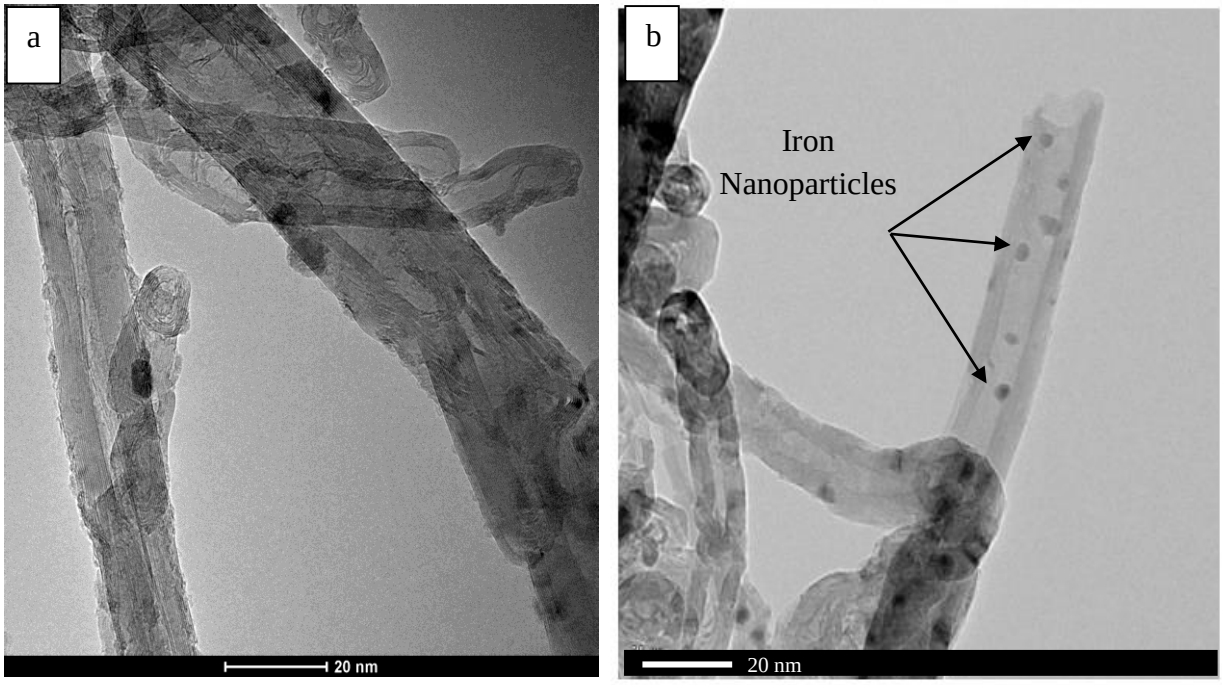

Figure 4:8 TEM images of (a) MWCNTs and (b) CNT-Fe_2O_3

4.2 Thermal Degradation Analysis

The study of the thermal oxidation of materials is of major importance. Since it can, in many cases, determine the upper temperature limit of use for a material. In addition, considerable attention has been directed towards the exploitation of thermogravimetric data for the determination of functional groups. For this purpose, thermogravimetric analysis (TGA) and derivative thermogravimetric (DTG) is a technique widely used because of its simplicity and the information afforded by a simple thermogram.

Figures 4.9 through 4.16 depict the TGA-DTG results for the doped carbon nanotubes with impregnation with metal oxide nanoparticles (CNTs-Fe_2O_3, CNT-CuO and CNT-Al_2O_3) and functionalized CNT with PEG.

It is clear from these figures, that the initial degradation of CNTs which was carried out in air condition started at approximately 550 °C and reached a maximum weight loss of at about 600 °C and completes at about 670°C as revealed by the DTG curve.

For CNTs impregnated with 1 wt. % iron nanoparticles the initial oxidation temperatures started at 500 °C reached a maximum weight loss of at about 550 °C and completed at about 600°C as revealed by the DTG curves shown in figure 4.10. While increasing the loading of iron oxide to 10 wt. % shifted the oxidation peak to lower value in which initial oxidation at this condition started at 450 °C with maximum weight losses at 500 °C and completed the oxidation at 540 °C. The same behavior was observed for CNTs impregnated with copper oxide nanoparticles as shown in figures 4.11 and 4.12. For

1wt.% of copper oxide nanoparticles, the oxidation temperature started at 500 °C and completed at 600 °C. While for 10 wt.% copper oxide the oxidation was completed at 540°C. However, for the impregnated CNT with 1wt.% aluminum oxide, the oxidation was competed at 680 °C and shifted to 700 °C for 10 wt.% aluminum oxide as shown in figures 4.13 and 4.14.

It is know that iron and copper oxide particles have high thermal conductivity, for this reason the CNTs doped with either iron oxide or copper oxide nanoparticles tend to burn faster than normal CNTs, due to the high transfer of the heat from the surface of the particles to the surface of CNTs.

Figures 4.15 and 4.16 depict the TGA-DTG results for the CNTs functionalized with PEG. In this case, the thermal degradation of functionalized CNT with PEG was carried out under nitrogen in order to degrade the PEG only. It is clear that the initial degradation temperature of functionalized CNT with PEG started at 300°C and complete degradation was at 420^{o}C, this is clearly shown in the DTG curve in figure 4.14. These two peaks at 320°C and 550°C in DTG curve (figure 4.15) were assigned to maximum degradation of poly ethylene glycol and carboxylic acid group.

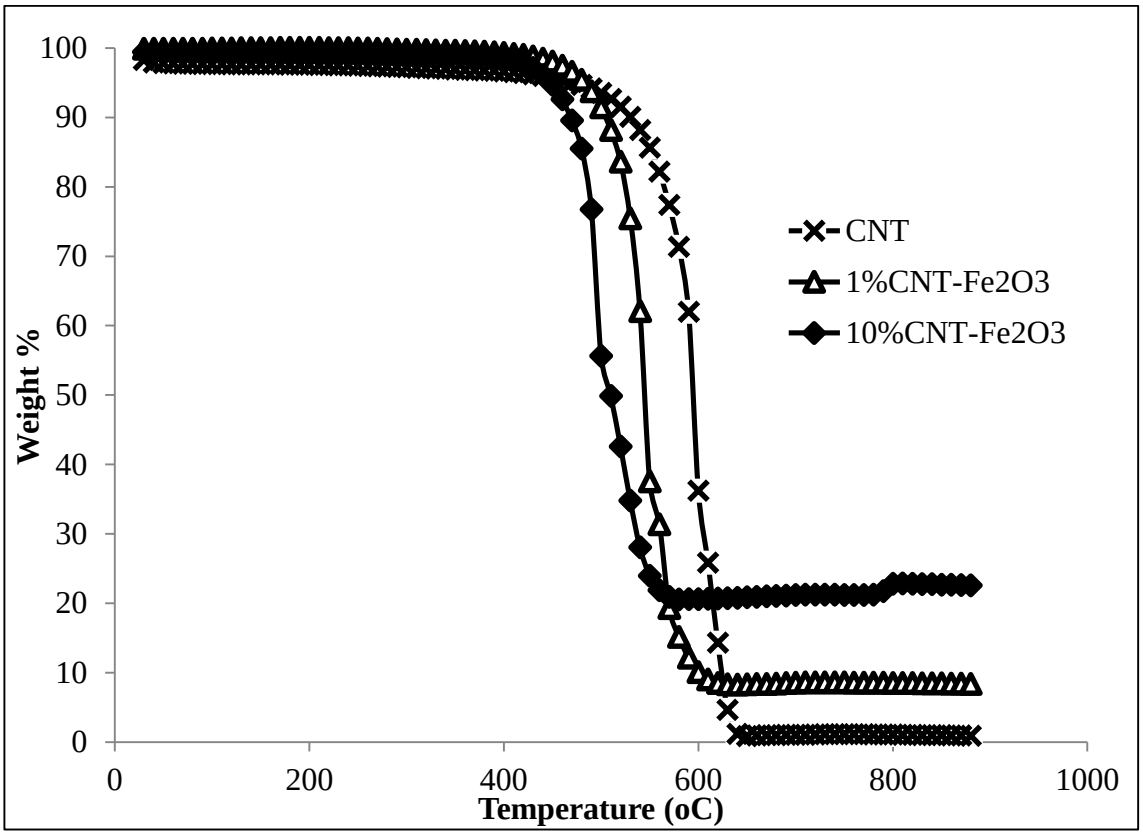

Figure 4:9 Thermogravimetric Analysis (TGA) for undoped and doped CNTs with iron oxide

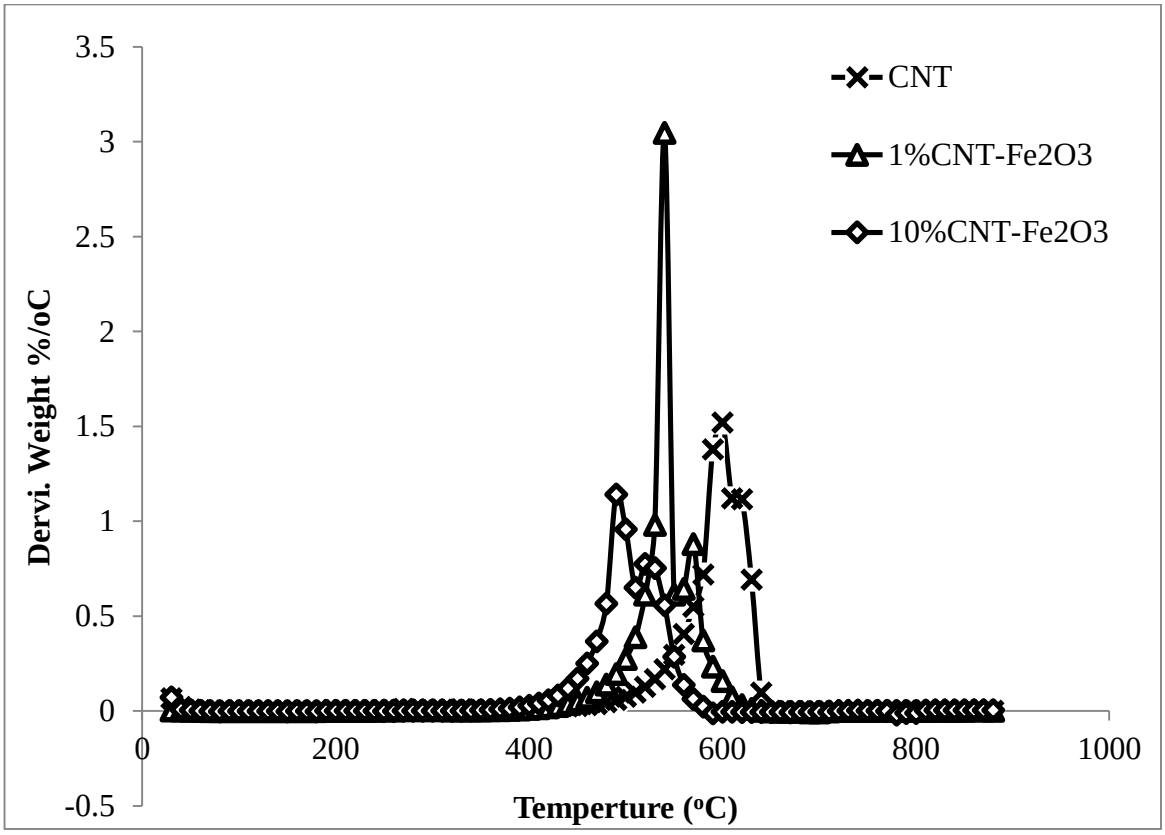

Figure 4:10 Thermogravimetric (DTG) for undoped and doped CNTs with iron oxide

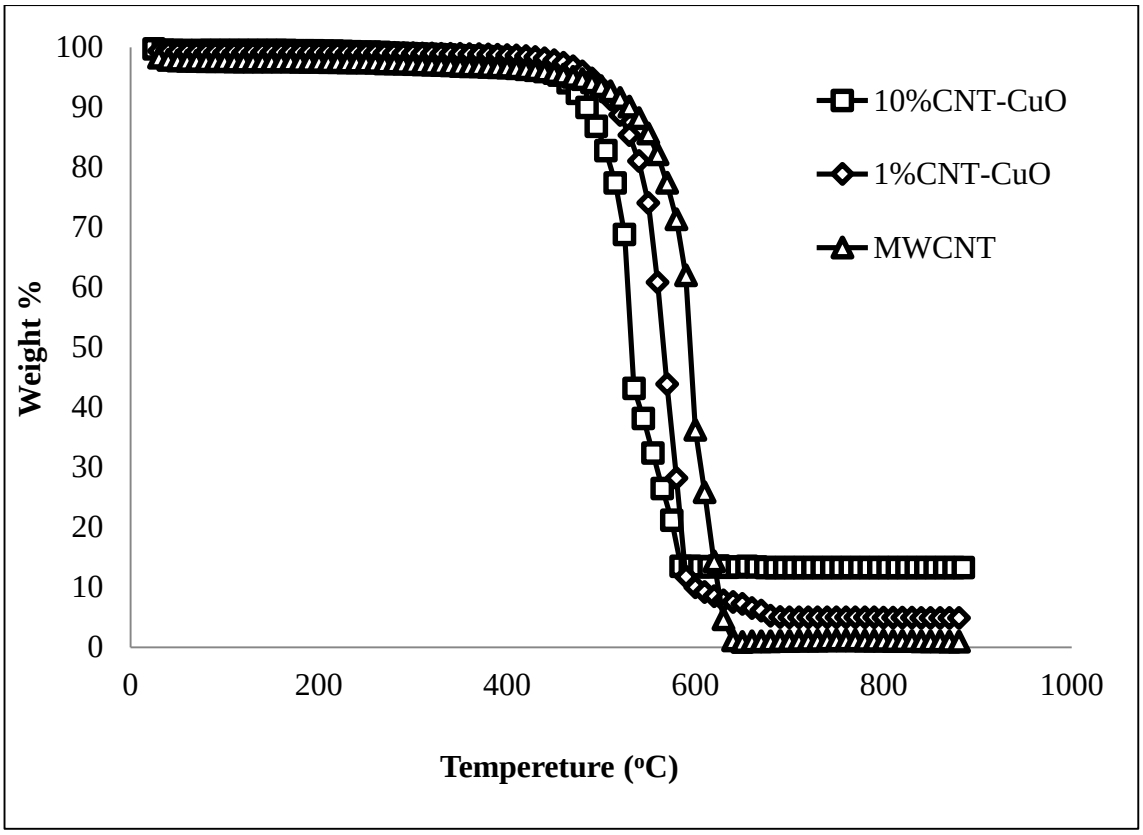

Figure 4:11 Thermogravimetric Analysis (TGA) for undoped and doped CNTs with copper oxide

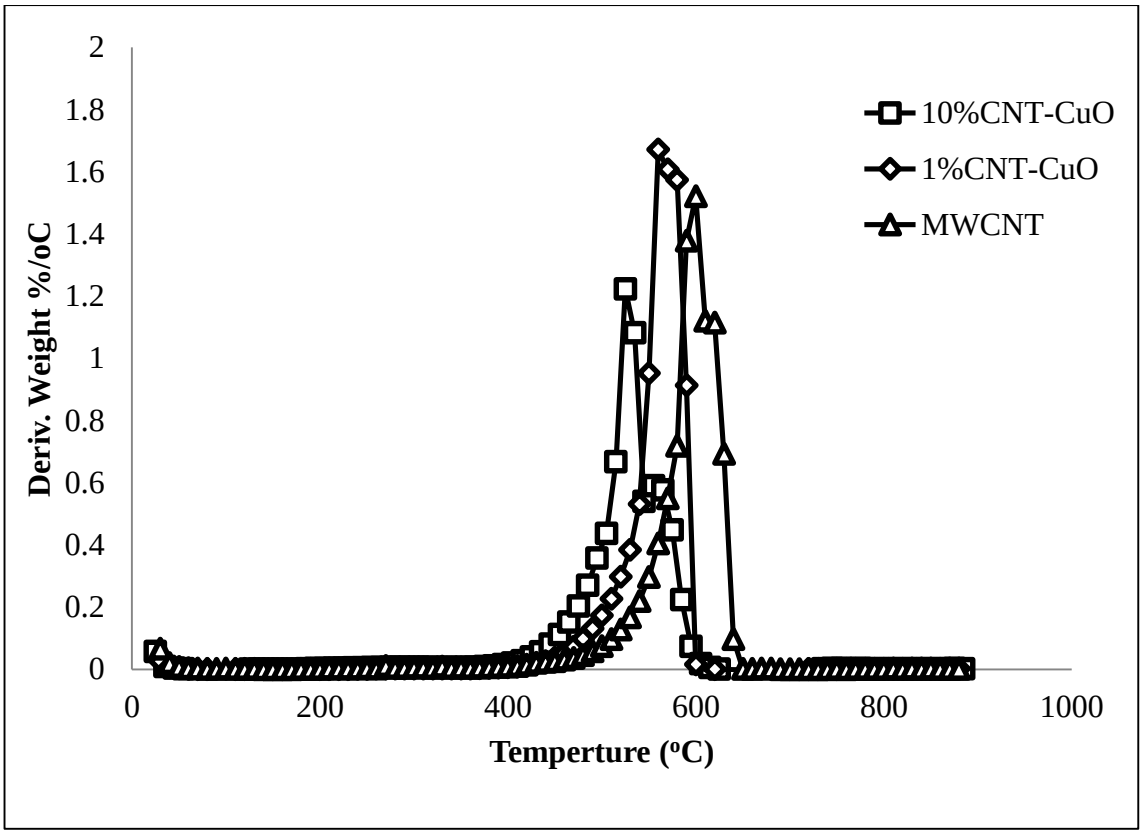

Figure 4:12 Thermogravimetric (DTG) for undoped and doped CNTs with copper oxide

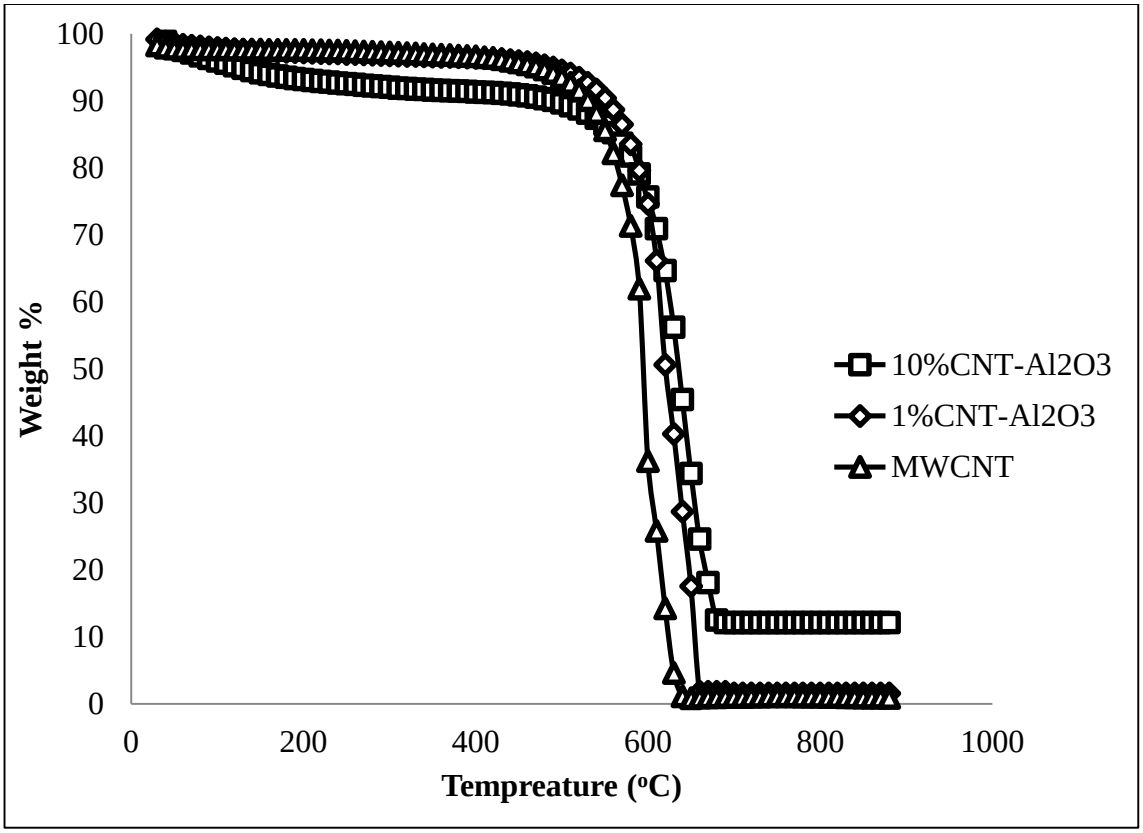

Figure 4:13 Thermogravimetric Analysis (TGA) for undoped and doped CNTs with aluminum oxide

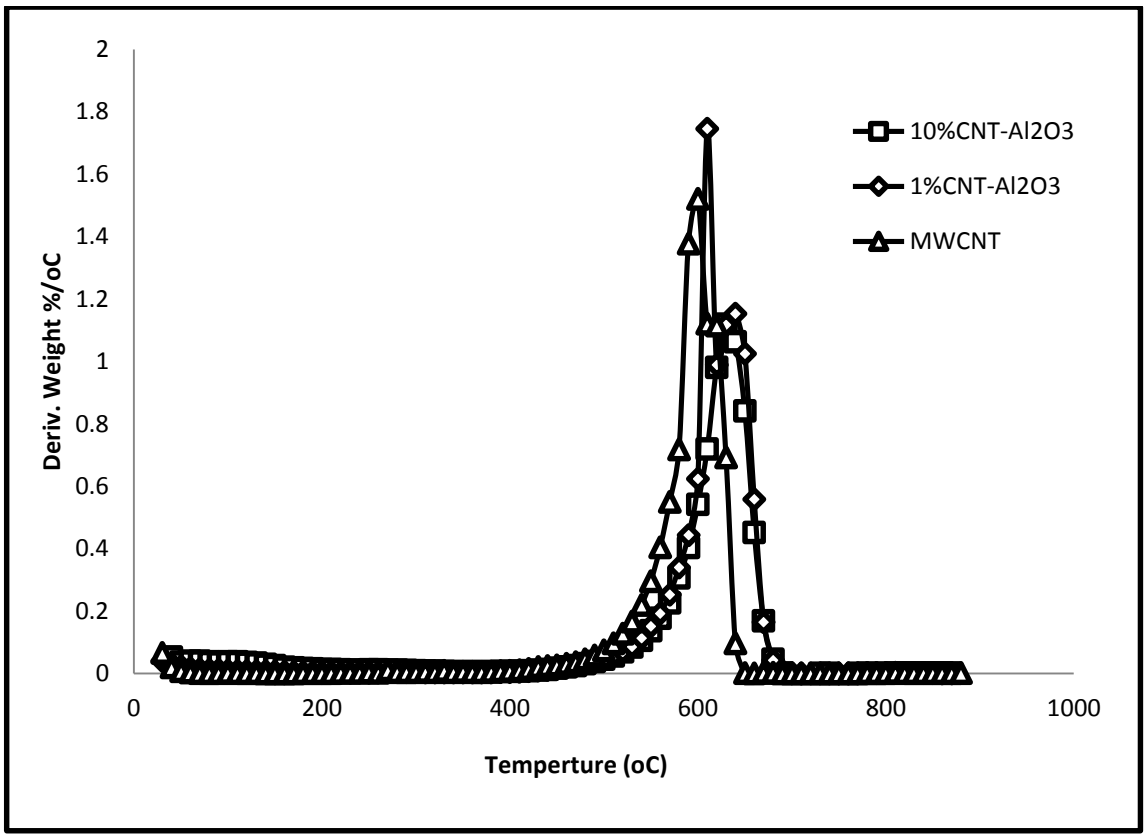

Figure 4:14 Thermogravimetric (DTG) for undoped and doped CNTs with aluminum oxide

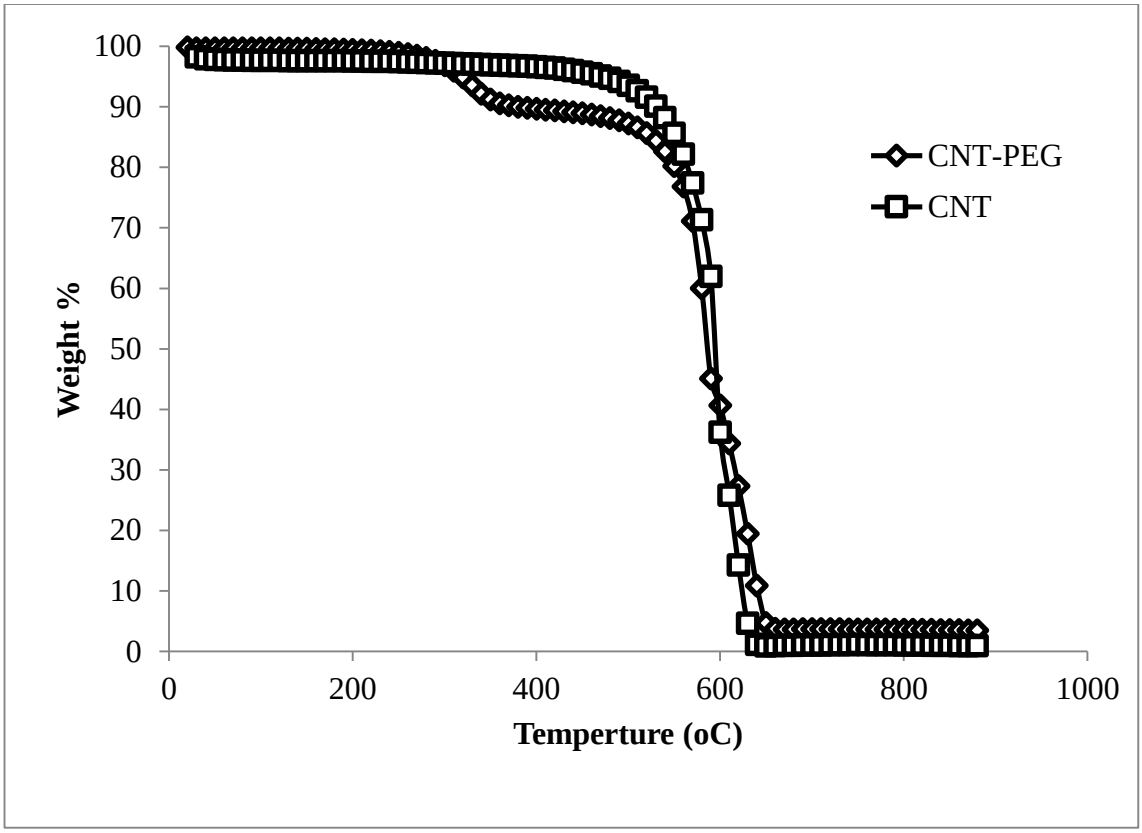

Figure 4:15 Thermogravimetric analysis (TGA) for CNTs and CNT-PEG

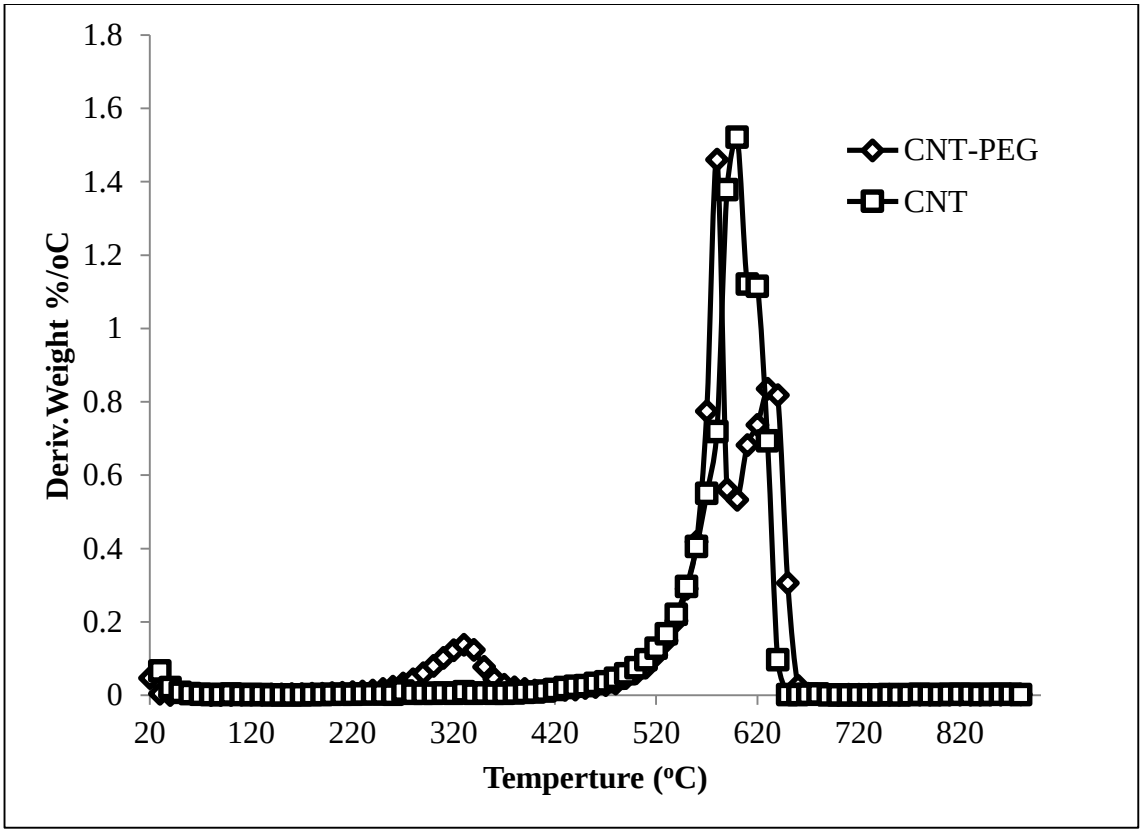

Figure 4:16 Thermogravimetric (DTG) for CNTs and CNT-PEG

4.3 Viscosity of nanofluids

Figure 4.17 shows the viscosity of nanofluids as a function of temperature at different loading of CNTs. The results indicate that the viscosity of nanofluids significantly increased with increasing in the concentration of the CNTs. However, the viscosity decreased as the temperature of the fluid increased. The microscopic view of the molecular level of the fluids can be used to determine the reasons which lead to the decrease in the viscosity of the fluid with the increase in temperature. The viscosity of the fluid depends on two major factors. The first factor is strength of the atomic bonding between the molecules which depends on the structure, shape and the size of the molecules. The second factor is kinetic energy of the molecules which is directly proportional to the temperature of the system. It can be concluded that, increasing the temperature of the fluid will weaken the atomic bonding between the molecules and increase their kinetic energy which will lead to a decrease in the viscosity.

Figures 4.18 and 4.19 show the viscosity of nanofluids as a function of temperature at different loading of doped CNT with iron oxide for 1 and 10wt%. The results showed that the viscosity of the nanofluid at different loading of CNTs /doped CNTs at 0.01 wt.%, 0.05 and 0.1 wt.% (%loading in water) increased the viscosity of the nanofluid up to 3 %, 3.2 % and 11 % respectively. As shown in figures 4.17, 4.18 and 4.19 there is no effect of the iron oxide nanoparticles doped on CNTs on the viscosity of the fluid, since the viscosity of the fluid is function of the temperature of the fluid and the concentration of the particles.

Similar phenomena of the viscosity were observed when CNTs were doped with copper and aluminum oxides. Figures 4.20 to 4.23 show the viscosity of nanofluids as a function of temperature at different loading of doped CNTs with copper oxide and aluminum oxide at 1 and 10wt. %. As discussed before the viscosity of the nanofluid increased with increasing amount of nanoparticles in the solution. Furthermore, similar trend was observed also in the viscosity when CNTs was functionalized with PEG. It can be concluded that, there is no effect of the types of nanoparticles doped on CNTs or the chemical groups functionalized onto the surface of CNTs on the viscosity of the fluid, since the viscosity of the fluid is only a function of the temperature and the particle concentrations.

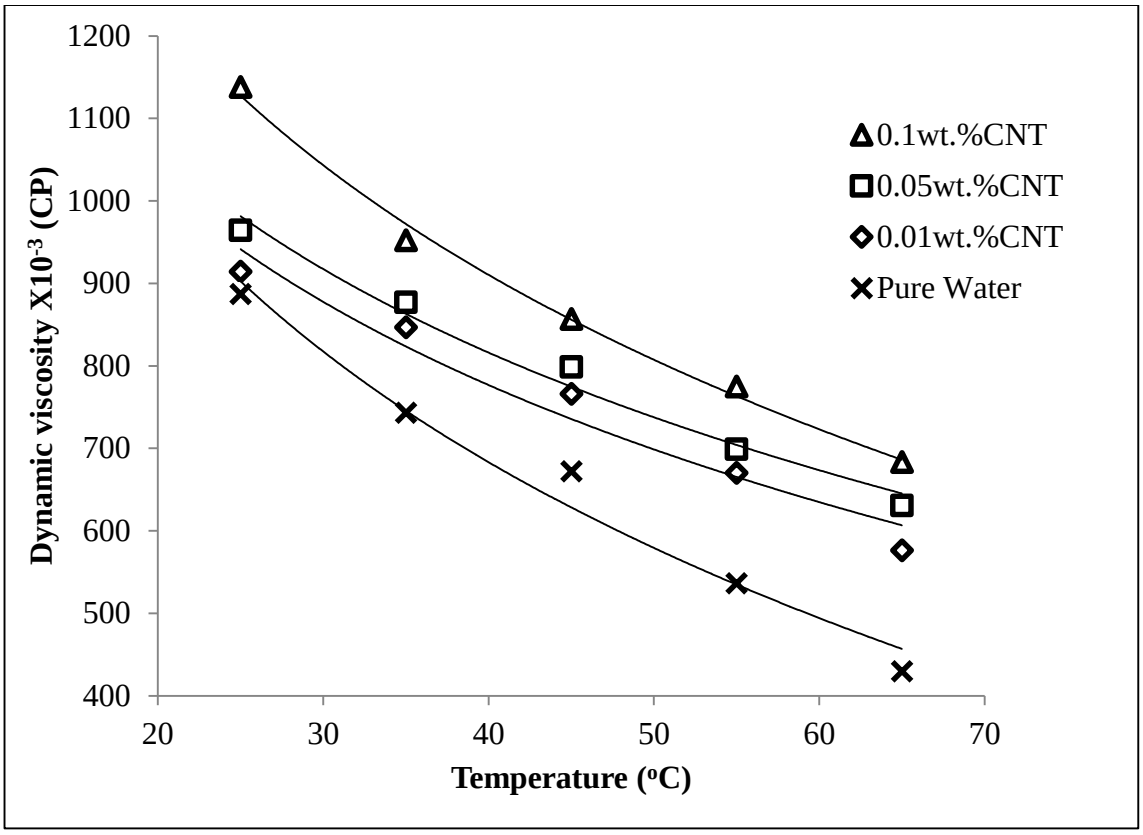

Figure 4:17 Variation of viscosity with respect to temperature for raw /undoped CNTs

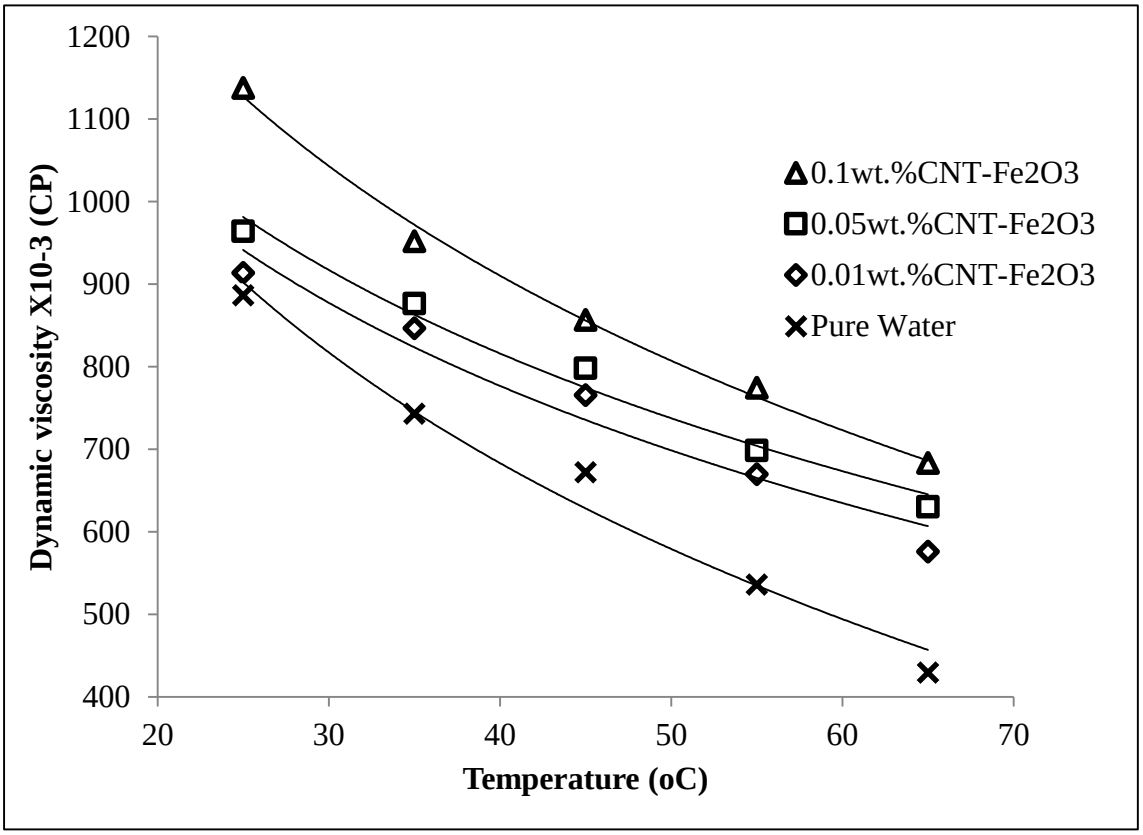

Figure 4:18 Variation of viscosity with respect to temperature for CNT-1 wt% Fe_2O_3.

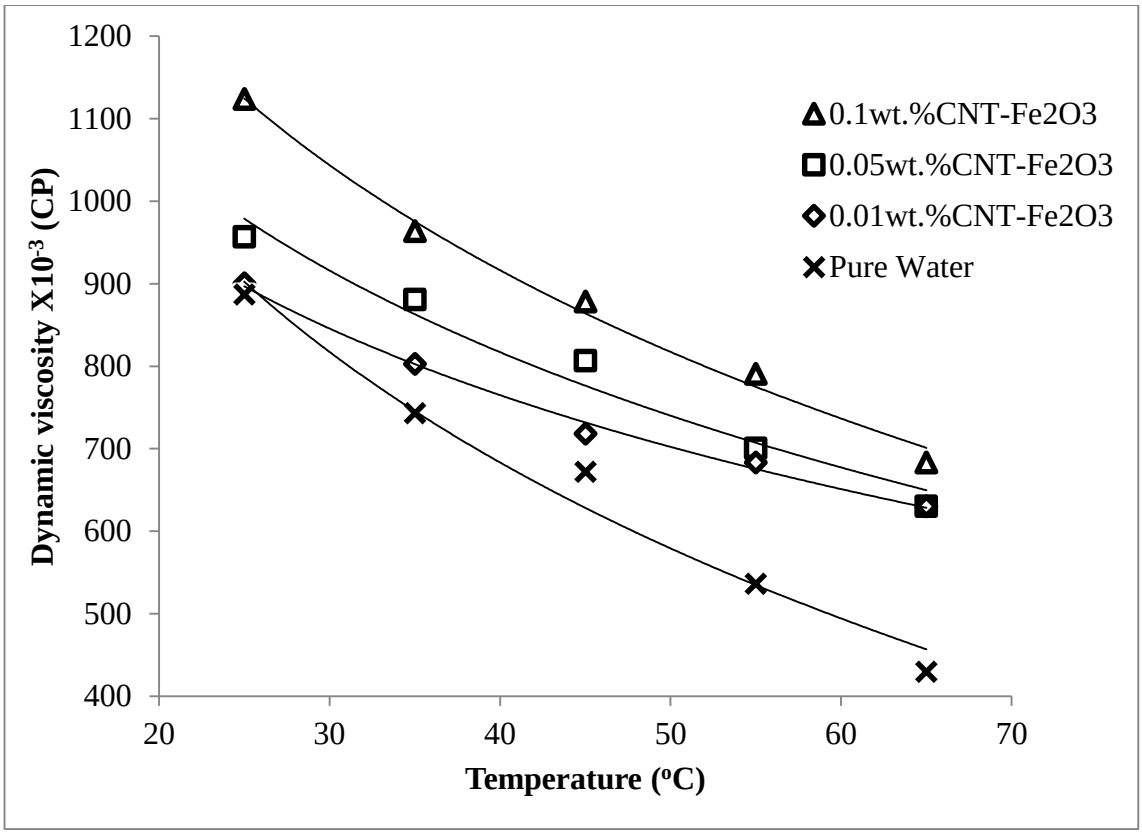

Figure 4:19 Variation of viscosity with respect to temperature for CNT-10 wt% Fe_2O_3.

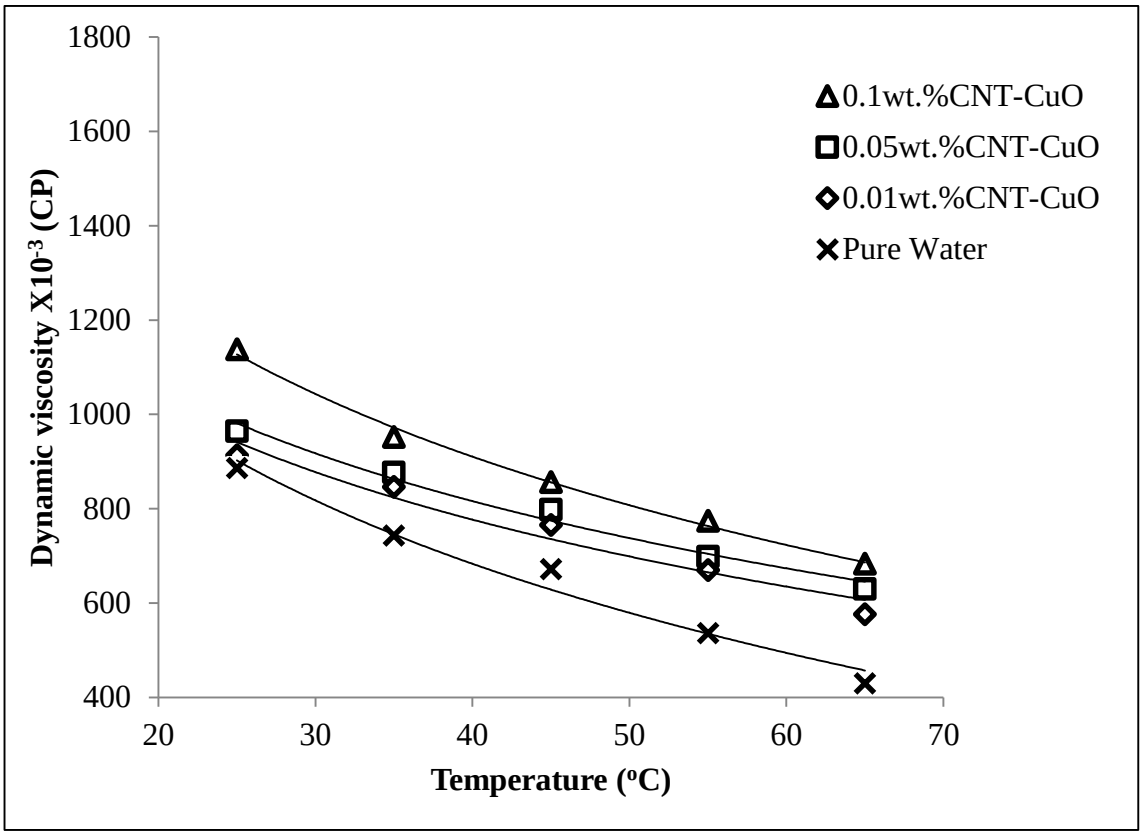

Figure 4:20 Variation of viscosity with respect to temperature for CNT-1 wt% CuO.

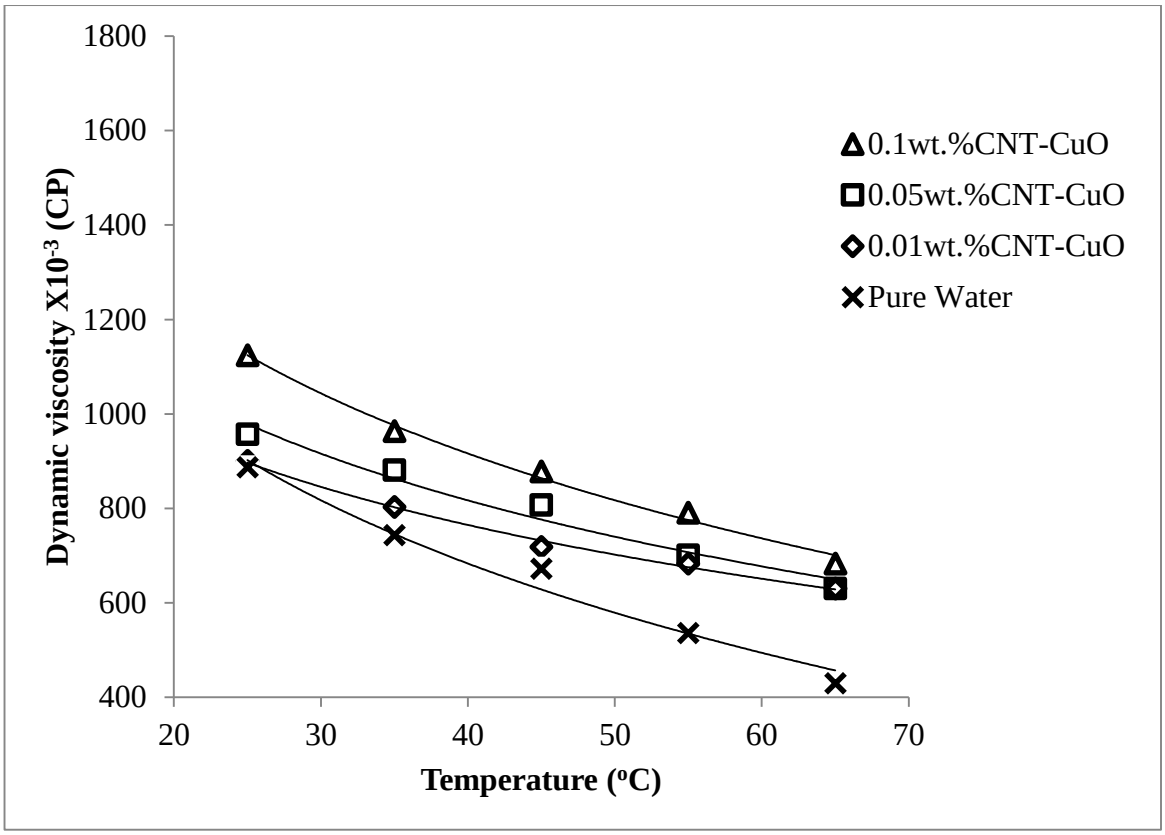

Figure 4:21 Variation of viscosity with respect to temperature for CNT-10 wt% CuO.

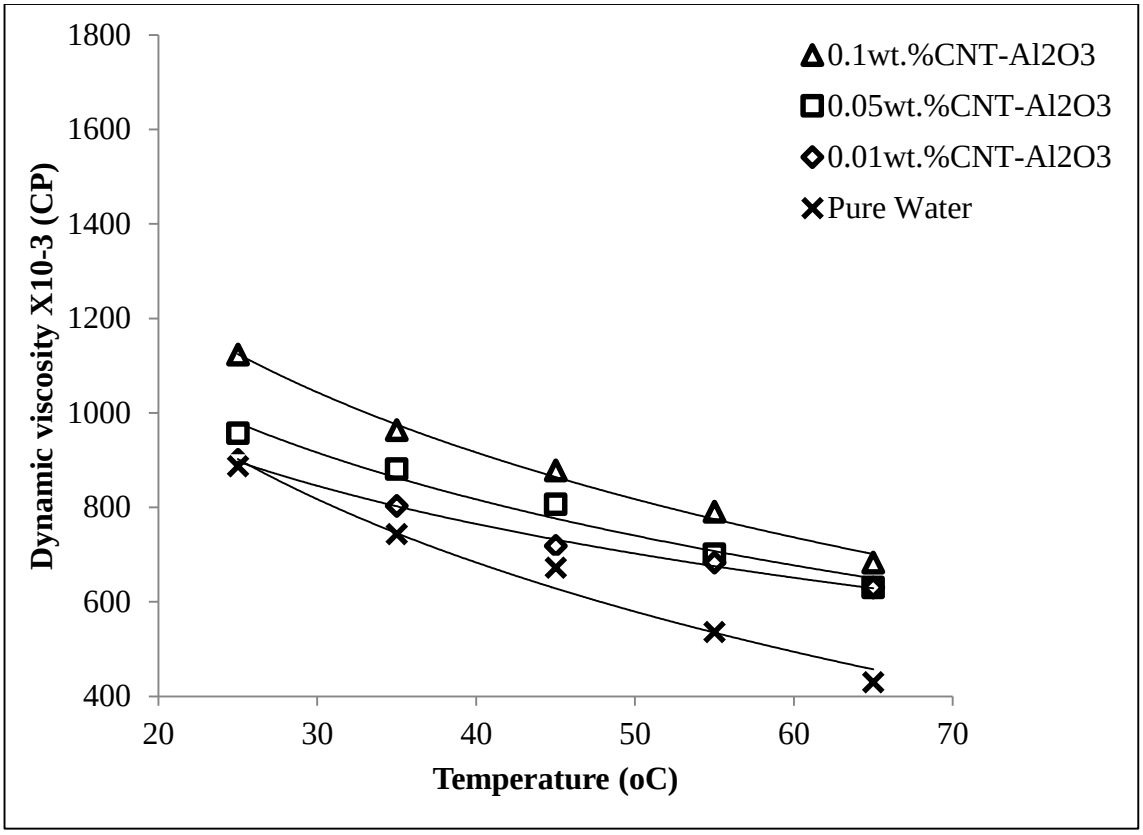

Figure 4:22 Variation of viscosity with respect to temperature for CNT-1 wt% Al_2O_3.

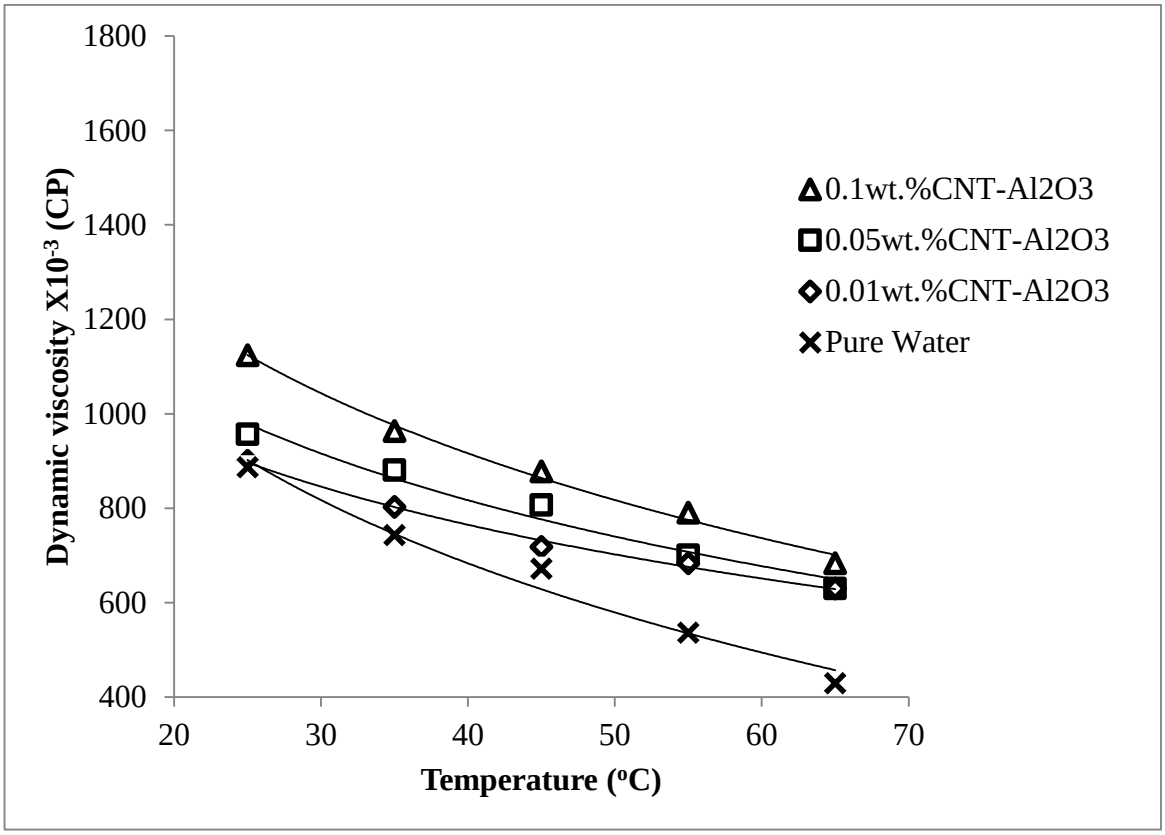

Figure 4:23 Variation of viscosity with respect to temperature for CNT-10 wt % Al_2O_3.

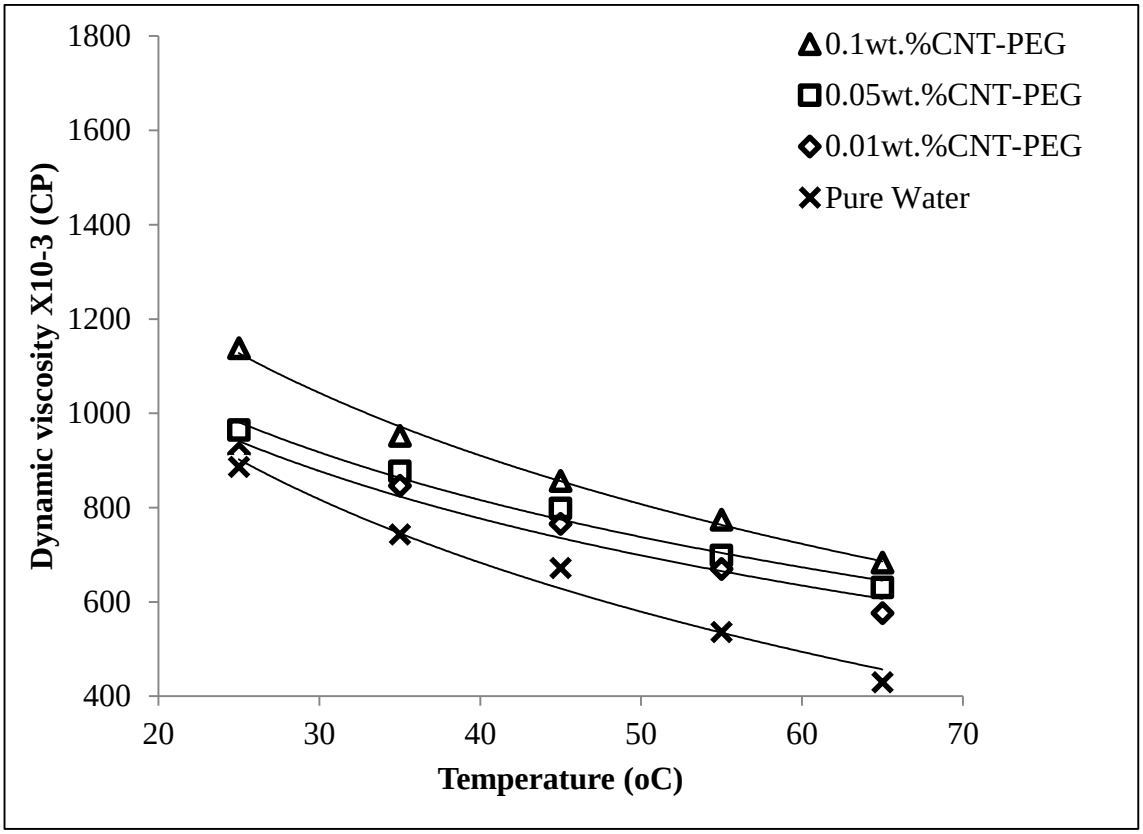

Figure 4:24 Variation of viscosity with respect to temperature for CNT-PEG.

4.4 Characterization of Functionalized Carbon Nanotubes with Polyethylene Glycol by FTIR

The spectrum of functionalized MWCNTs was analyzed using FT-IR instrument. The sample was prepared using potassium bromide at 0.03 wt.% concentration before FTIR measurements.

Figure 4.25 shows the FT-IR spectrum of MWCNTs functionalized with polyethylene glycol (PEG) compared with modified/raw CNTs. The FTIR spectra for MWCNTs exhibited a broad peak at 3431 cm^{-1}, which is refered to the O-H stretching of the hydroxyl group in PEG. The peak at 3000-2900cm^{-1} can be attributed to symmetric and asymmetric C–H stretching of methylene group in polyethylene glycol. While the new peak at wave number of 1086cm^{-1} is due to the C–O stretch of ester between oxide MWCNTs and PEG. This indicates that the MWCNTs were indeed functionalized with PEG.

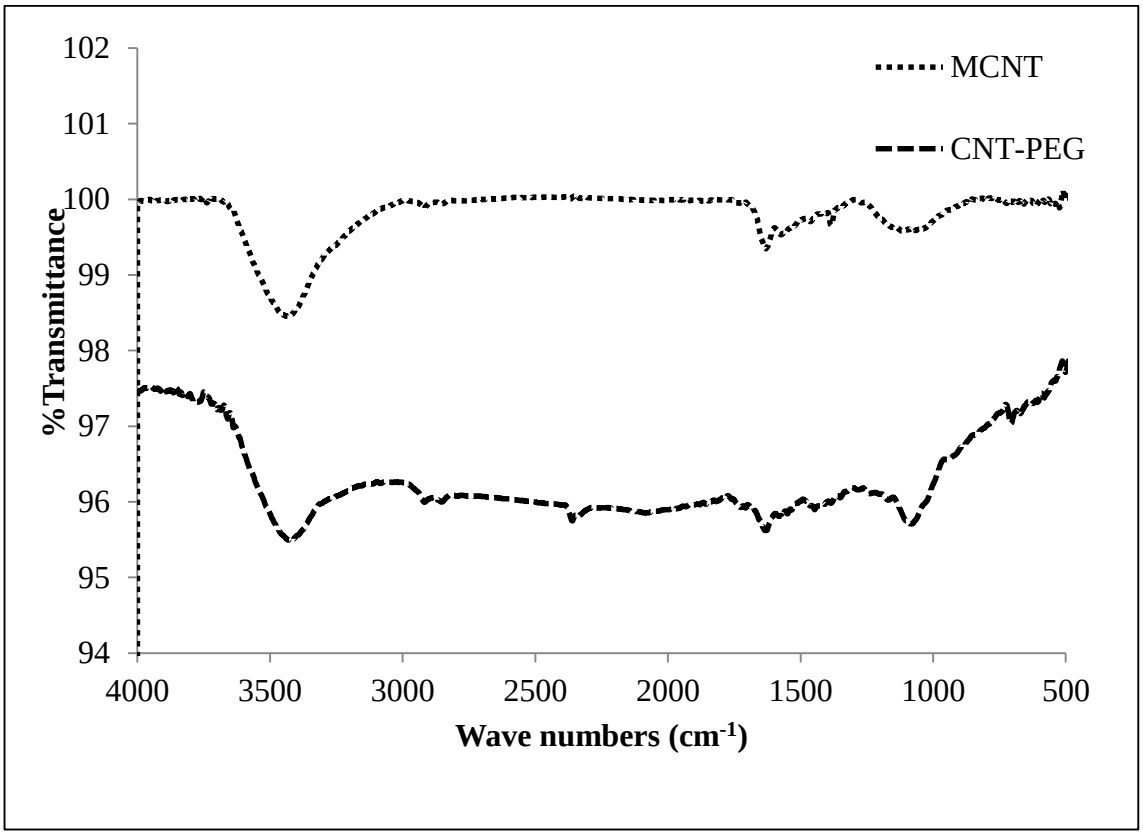

Figure 4:25 The FTIR spectrum of functionalized CNT with polyethylene glycol and raw/modified CNT

4.5 Specific heat capacity of nanofluids:

The heat capacity of water as base fluid mixed, with different concentration (0.01, 0.05 and 0.1wt.%) of unmodified and modified CNTs with metal oxide nanoparticles such as iron oxide; copper oxide and aluminum oxide at (1 and 10% wt.%) and functionalized with polyethylene glycol (PEG) functional groups, was investigated using DSC technique from 25^{o}C to 50^{o}C at heating rate of 1.5^{o}C/min.

Figures 4.26 shows the enhancement of the heat capacity of nanofluids ($Cp,_{nf}/Cp_{w}$) as a function of temperature. Where $Cp,_{nf}$ is the specific heat capacity of nanofluids and Cp_{w} is the specific heat capacity of water. The results showed that the heat capacity of nanofluids increased significantly with an increase in the concentration of undoped CNTs. The maximum enhancement of the specific heat for unmodified CNTs was 8%, while for the modified CNTs with (1 wt. % Fe_2O_3 and 10 wt. % Fe_2O_3) at weight concentration of 0.1 wt.% and 35^{o}C, the enhancement was 19 % and 38 % respectively as shown in figures 4.27 and 4.28. It can be noted that the unmodified CNTs enhanced slightly the heat capacity of the water, while impregnated or modified CNTs with iron oxide nanoparticles increased dramatically the heat capacity of water. It has been reported by different research groups [57] that the specific heat capacity of the CNTs is ranging from 0.6-0.7 (J/g.K) while the specific heat capacity of iron oxide particles is 0.451 (J/g.K) [57]. Based on these values the CNTs required 6 times less energy to increase the temperature by 1 ^{o}C compared to water. For CNTs doped with iron particles, it requires 9 times less energy than water. Mixing these highly thermally conductive nanoparticles with water will

defiantly enhance the thermal properties of the nanofluid as observed from these results, which are in agreement with TGA results, as mentioned above where CNTs doped with iron nanoparticles burned faster than normal due to high heat transfer of iron nanoparticles.

Figures 4.29 and 4.30 show the enhancement of the heat capacity of doped CNT with copper oxide nanofluids ($Cp_{,nf}/Cp_w$) as a function of temperature. As it can be seen, the heat capacity of nanofluids increased significantly with an increase in the concentration of CNT-CuO and also slightly increased with temperature. The maximum enhancement of the specific heat for modified CNTs with (1 wt. % CuO and 10 wt. % CuO) at weight concentration of 0.1 wt. % and 35^{o}C was 20 % and 49 % respectively. It can be noted that the unmodified CNTs enhanced slightly the heat capacity of the water, while impregnated or modified CNTs with copper oxide nanoparticles increased dramatically the heat capacity of water. The same phenomena was observed for the doped CNT with 1 wt% Al_2O_3 and 10 wt% Al_2O_3 as shown in figures 4.31 and 4.32. The maximum enhancement of the specific heat for modified CNTs with (1 wt % Al_2O_3 and 10 wt % Al_2O_3) at weight concentration of 0.1 wt % and 35^{o}C was 33 % and 50 % respectively.

The specific heat capacity was also measured for the functionalized CNTs with PEG, as shown in figure 4.33. The heat capacity of nanofluids intensely increased with an increase in the concentration of functionalized CNTs with PEG. The maximum enhancement in the specific heat of functionalized CNTs with PEG at weight concentration of 0.1 wt % and 35^{o}C was 45 %. It can be noted that the raw CNTs enhanced slightly the heat capacity of the water, while functionalized CNTs with PEG increased drastically the heat capacity of water.

It has been reported by different research groups that there are many mechanisms to explain the enhancement in heat transfer of nanofluids. They are based on aggregation of nanoparticles [22], Brownian motion [57] and size and shape of nanoparticles.

The larger surface area can thus increase heat transfer capabilities [59][47]. The fluids containing suspended solid particles in a nano scale level have better heat capacities than the fluids containing coarse solid particles on a micro scale. Furthermore, in 2005 Chon et al. [63] confirmed the effect of temperature on thermal conductivity, and they also found the enhancement of thermal conductivity in nanofluids based water using alumina nanoparticles was inversely proportional with particle size.

The motion of nanoparticles in fluids acts as agitator and has an important role in the enhancement of the heat capacity. So the Brownian motion of particles is the key mechanism of the thermal conductivity enhancement in nanofluids.

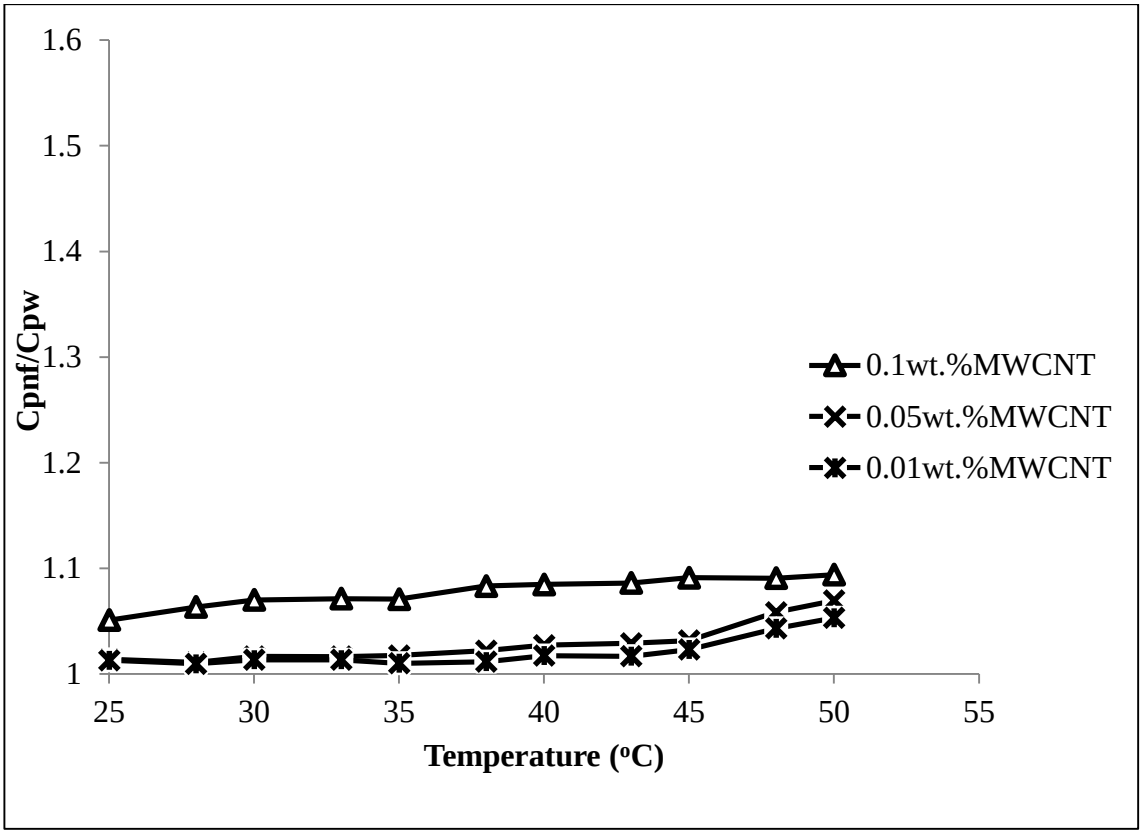

Figure 4:26 Enhancement in heat capacity of raw CNTs nanofluids with respect to temperature.

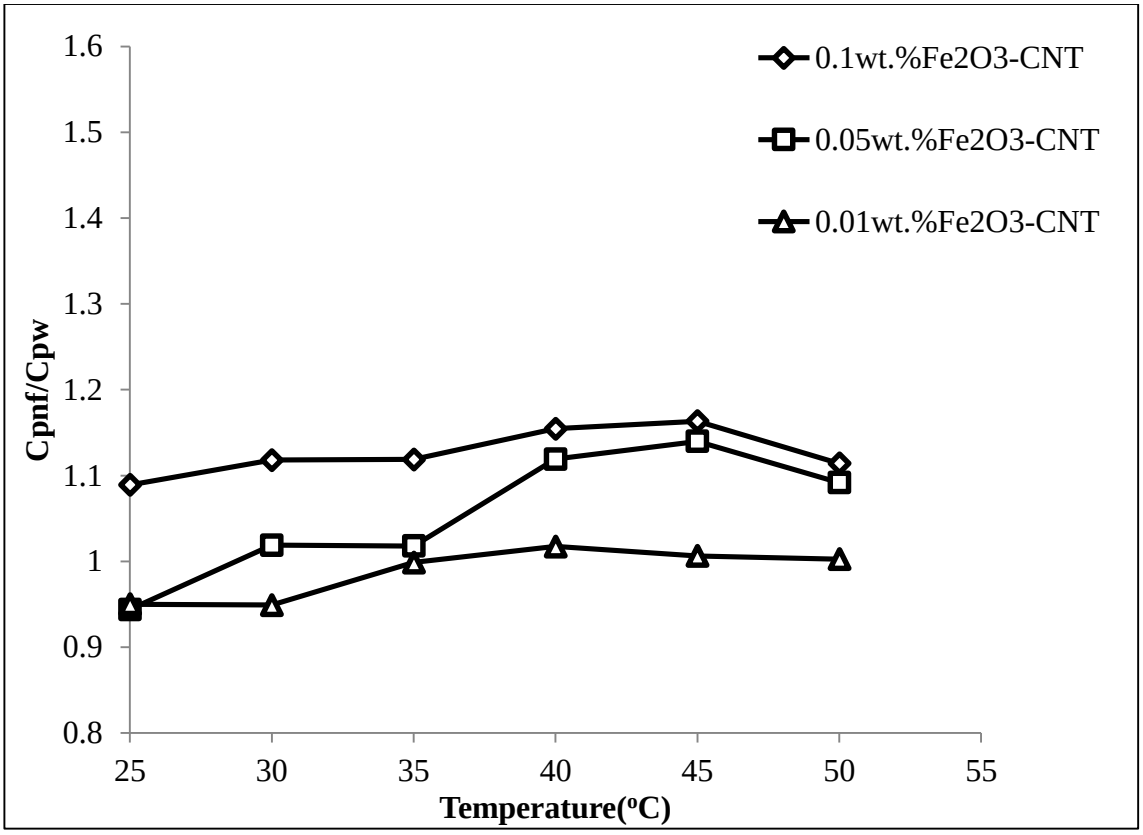

Figure 4:27 Enhancement in heat capacity of CNT- 1 wt% Fe_2O_3 nanofluids with respect to temperature.

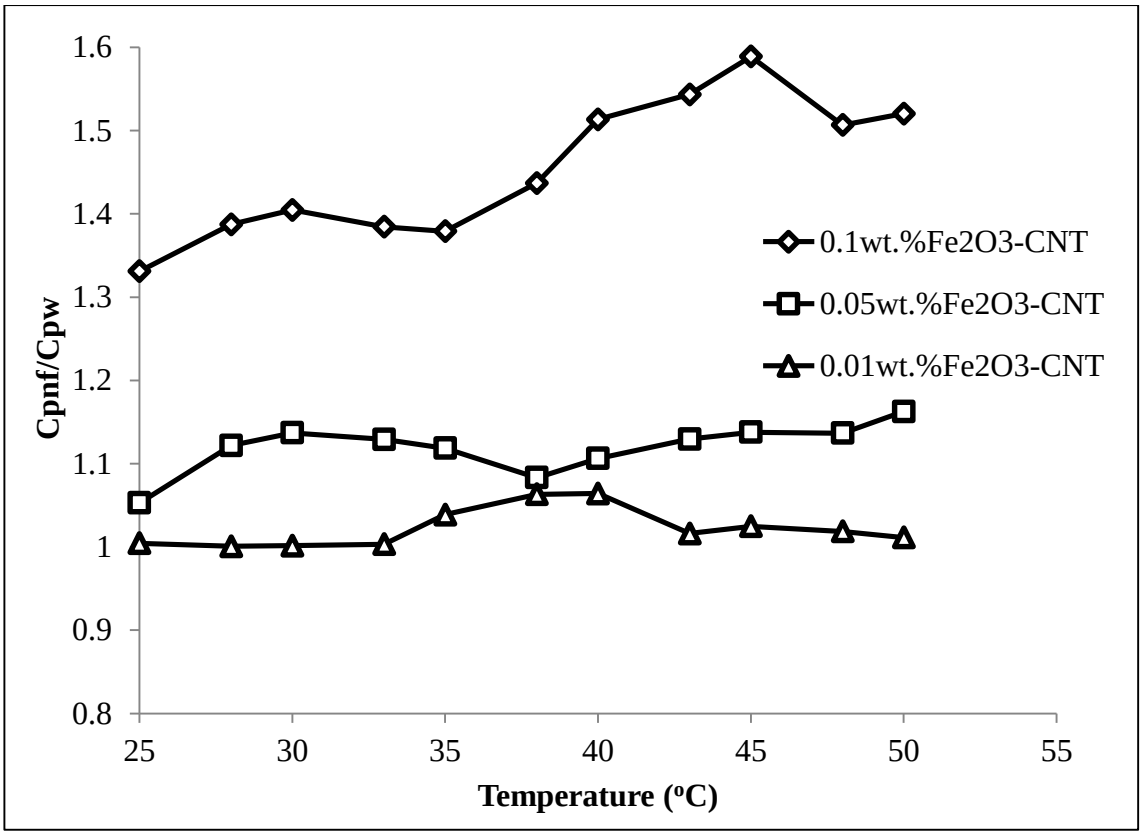

Figure 4:28 Enhancement in heat capacity of CNT- 10 wt% Fe2O3 nanofluids with respect to temperature.

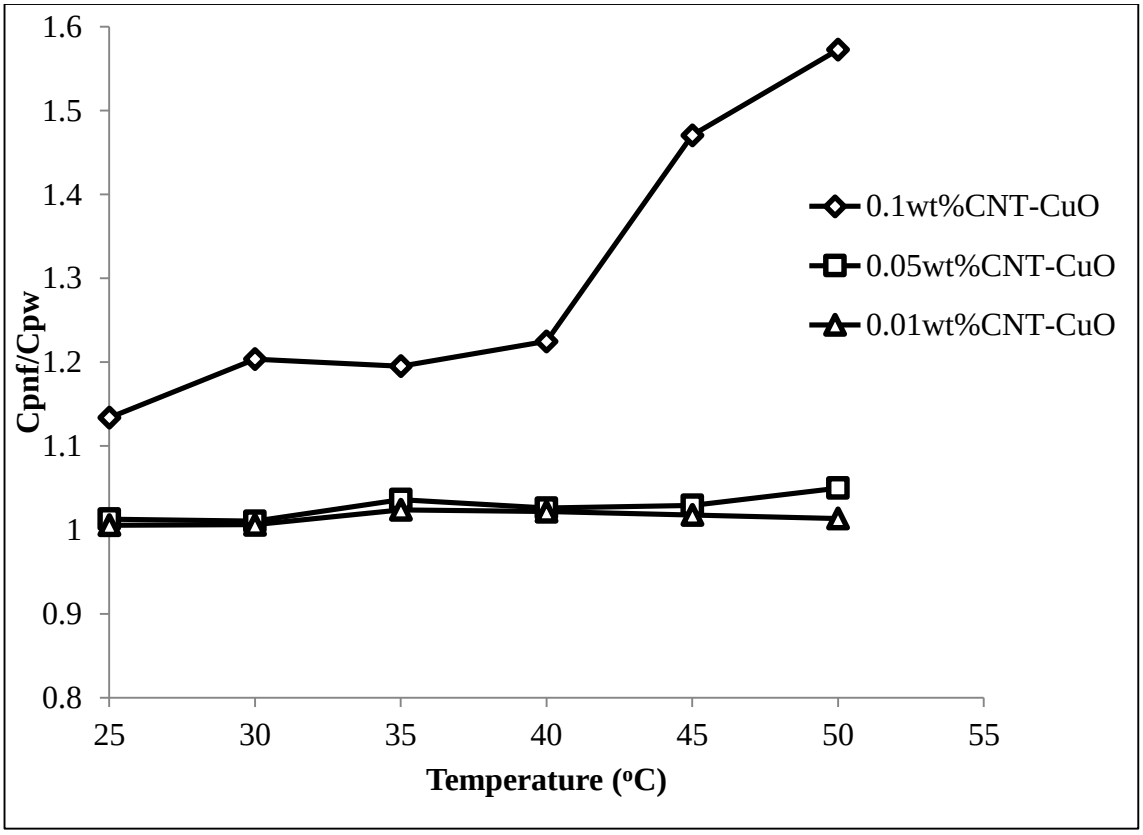

Figure 4:29 Enhancement in heat capacity of CNT- 1 wt % CuO nanofluids with respect to temperature.

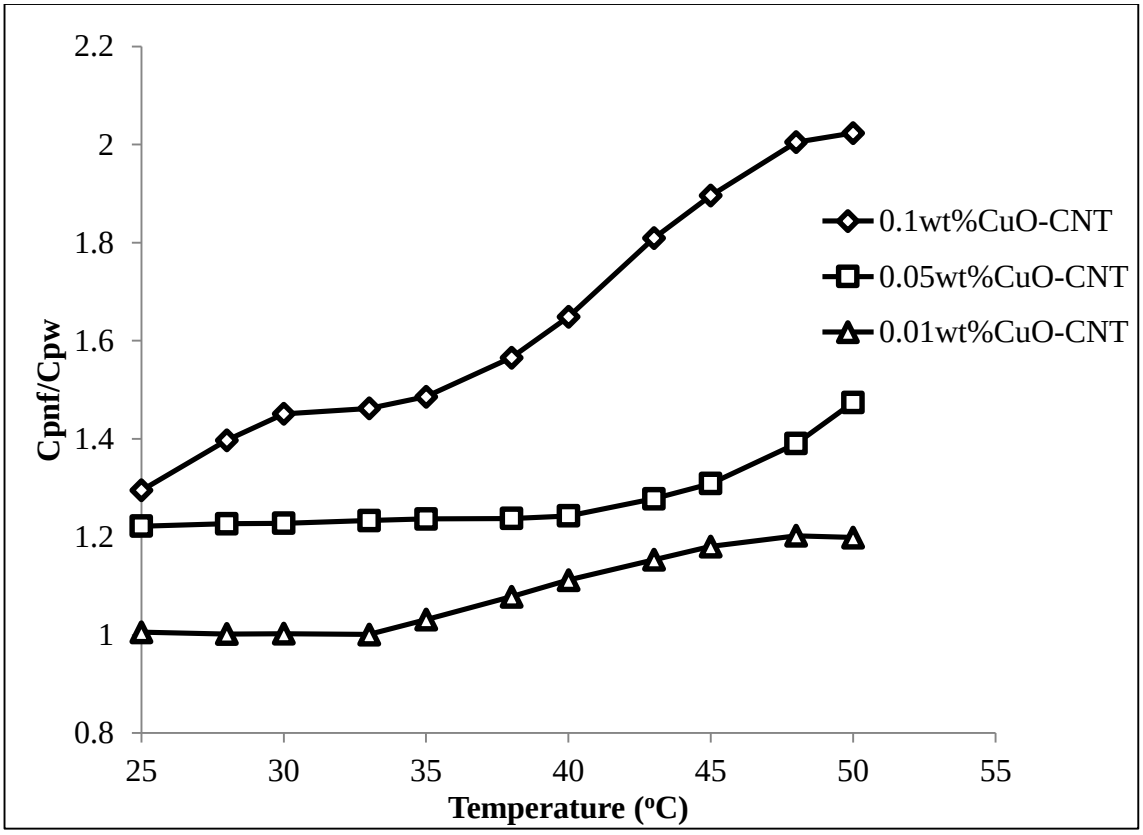

Figure 4:30 Enhancement in heat capacity of CNT- 10 wt % CuO nanofluids with respect to temperature.

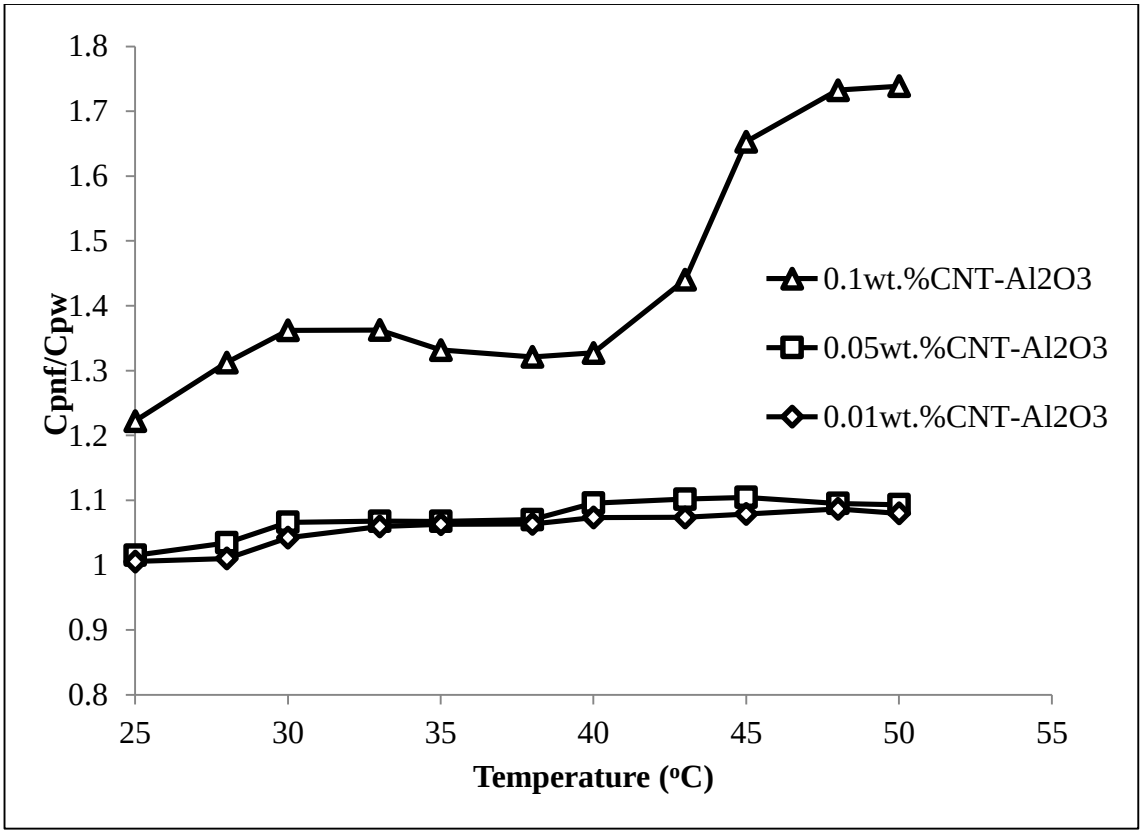

Figure 4:31 Enhancement in heat capacity of CNT- 1 wt % Al_2O_3 nanofluids with respect to temperature.

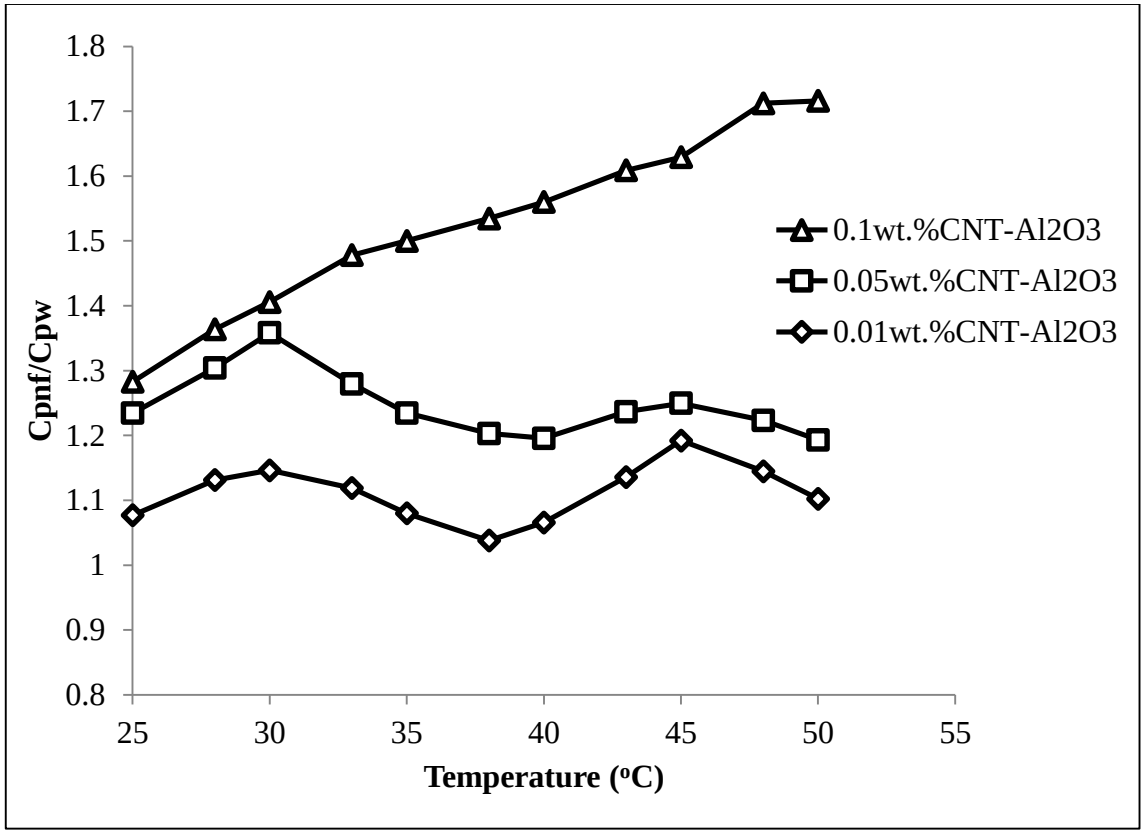

Figure 4:32 Enhancement in heat capacity of CNT- 10 wt % Al2O3 nanofluids with respect to temperature.

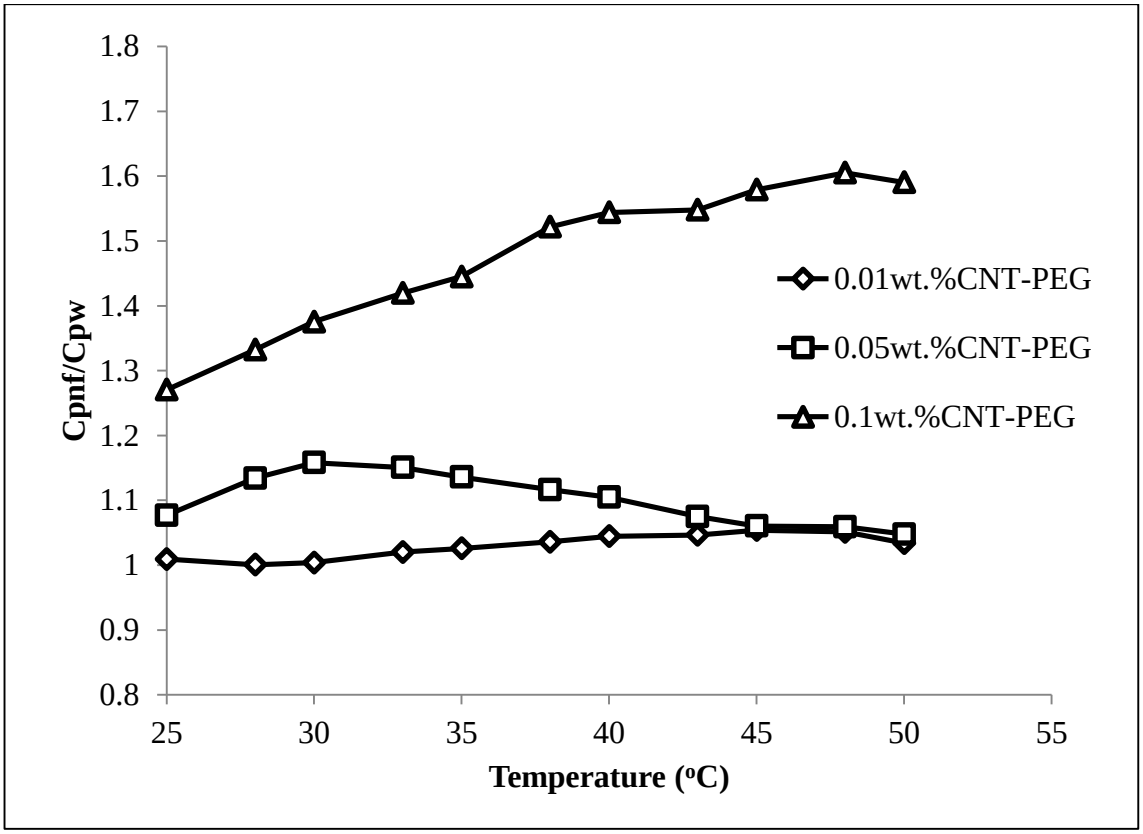

Figure 4:33 Enhancement in heat capacity of CNT-PEG nanofluids with respect to temperature.

4.6 Heat Transfer of Nanofluids

Several experiments were conducted to investigate the heat transfer of nanofluids using a single tube heat exchanger. Five types of nanomaterial unmodified CNTs, and modified CNTs with metal oxide (iron oxide, copper oxide and aluminum oxide) with 1 wt. % and 10 wt.% loading and functionalized CNT with PEG were used in this work to investigate their effect on the heat transfer of the nanofluid. The nanofluids were prepared at three weight concentrations of nanomaterial (0.01, 0.05 and 0.1wt. %). The inlet temperature of the nanofluid was fixed by controlled heating bath at 35 ^{o}C while their flow rate was controlled by digital mass flow controller to be at turbulent regime ranging from 200 -640 kg/h.

Figure 4.34 shows the effect of different weight concentration of CNTs (0.01, 0.05 and 0.1wt. %) on the enhancement of the heat transfer of the nanofluids at different flow rates (kg/h). It was observed that the heat transfer ratio ($Q_{nanofluids}/Q_{water}$) increased with an increase in the weight concentrations of unmodified CNTs. The maximum enhancement of heat transfer was 15 % at 0.1 wt % and 400 kg/h mass flow rate. This result is consistent with the Cp results of the CNTs-water nanofluid which showed enhancement of the heat capacity of water by about 8 % after adding the CNTs. The other 7 % enhancement in the heat transfer of nanofluid could be from the enhancement of the dispersion of the CNTs into the solution due to high motion of the fluid which reduced the agglomeration and increased the dispersion. The agglomeration of the nanoparticles is one of the major problems that makes the fluid unstable. In order to have a stable fluid, the particles have to be dispersed in the solution either mechanically or chemically. Mechanically by applying sonication waves to break down the van der walls interaction

forces between the particles. Chemically is by modifying the surfaces of the particles by adding surfactant that is soluble in the fluid. In this study only the mechanical approach was used in order to prevent the effect of the chemical surfactant on the properties of the nanofluids which might affect the viscosity, the heat capacity and the chemical structure of the fluid.

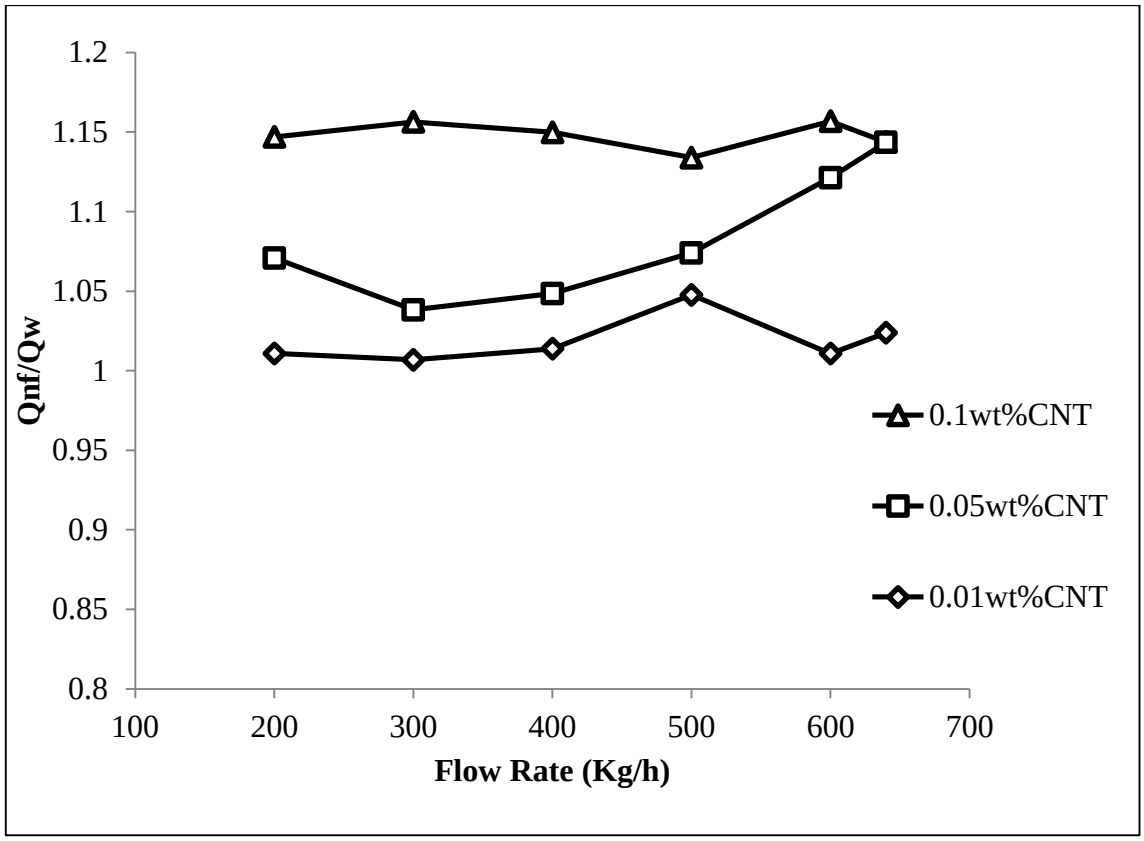

Figure 4:34 Enhancement heat transfer of nanofluid by different concentration of CNTs at different flow rates.

The same phenomena were observed when doped CNTs with iron nanoparticles were used. The heat transfer of the nanofluid increased with the increase in the concentration of doped CNTs as shown in figures 4.35 and 4.36. Due to the large enhancement in the heat capacity of the CNTs/ iron oxide composite, the heat transfer of the nanofluid containing CNTs/ iron oxide composite at 0.1 wt% increased by almost 30 % compared to water as base fluid and 15 % higher than undoped CNTs with water at the same condition. Increasing the loading of the iron oxide nanoparticles on the surfaces of CNTs to 10 wt % increased the heat transfer of the nanofluid up 60 % at the concentration of 0.1 wt%.

Figures 4.37 and 4.38 show the effect of different weight concentration of CNT impregnated with copper oxide (0.01, 0.05 and 0.1wt. %) on the enhancement of the heat transfer of the nanofluids at different flow rate (kg/h) for 1wt% and CNT-10wt.% CuO respectively. It is clear that the heat transfer of the nanofluid increased with the increase in the concentration of doped CNTs. Due to the large enhancement on the heat capacity of the CNTs/ copper oxide composite, the heat transfer of the nanofluid containing CNT-1wt.% CuO composite at 0.1 wt% increased by almost 23 % compare to water as base fluid and 8 % higher than undoped CNTs with water at the same condition. Increasing the loading of the copper oxide nanoparticles on the surfaces of CNTs to 10 wt. % increased the heat transfer of the nanofluid up to 58 % at the concentration of 0.1 wt.%. These results are consistent with the Cp results of the 1wt.% and CNTs-10wt.% Cuo-water nanofluid which showed enhancement of the heat capacity of water by 20 % and 49% respectively after adding the doped CNTs.

The same phenomena were observed when doped CNTs with aluminum nanoparticles were used. The heat transfer of the nanofluid increased with the increase in the concentration of doped CNTs as shown in figures 4.39 and 4.40. The maximum heat transfer of the nanofluid containing /CNT-1wt.% aluminum oxide composite at 0.1 wt. % was 44 %. Increasing the loading of the aluminum oxide nanoparticles on the surfaces of CNTs to 10 wt. % increased the heat transfer of the nanofluid up 57 % at the concentration of 0.1 wt. %. These results are consistent with the Cp results of the 1wt. % and CNTs-10wt.% aluminum oxide-water nanofluid which showed enhancement in the heat capacity of water by 33 % and 50% respectively after adding the doped CNTs.

The heat transfer of nanofluids using functionalized CNTs with PEG was also investigated with different concentrations (0.01wt.%, 0.05wt.% and 0.1wt.% CNT-PEG). As shown in figure 4.41, it was observed that the heat transfer ratio increased with an increase in the weight concentrations of CNT-PEG. The maximum enhancement in heat transfer was 44% at 500 kg/h. However, the minimum enhancement was 5% for 0.01 weight fraction at 200 kg/h.

It could be emphasized that the observed enhancement in heat transfer is much higher than that of the increase in heat capacity. One possible explanation could be the enhancement in the dispersion of the doped CNT in the solution, this due to high motion of the fluid which reduced the agglomeration and increased the dispersion during the flowing along the tube. Apart from the foregoing explanation, it should be highlighted that the measurement of specific heat capacity was performed under static condition, whereas the measurement of heat transfer rate was conducted at dynamic flow condition. Hence, interactions of the flow field with nanoparticles may be another reason for

substantial rise in heat transfer rate [47]. Other possible reasons for the enhancement in heat transfer of fluid by suspending the CNT could be firstly the nanoparticles led to increase in the surface area, thermal conductivity and heat capacity of the fluids. Secondly, the collision and interaction between the nanoparticles, water, mixing fluctuation and turbulence of the fluid were strengthened. [22]

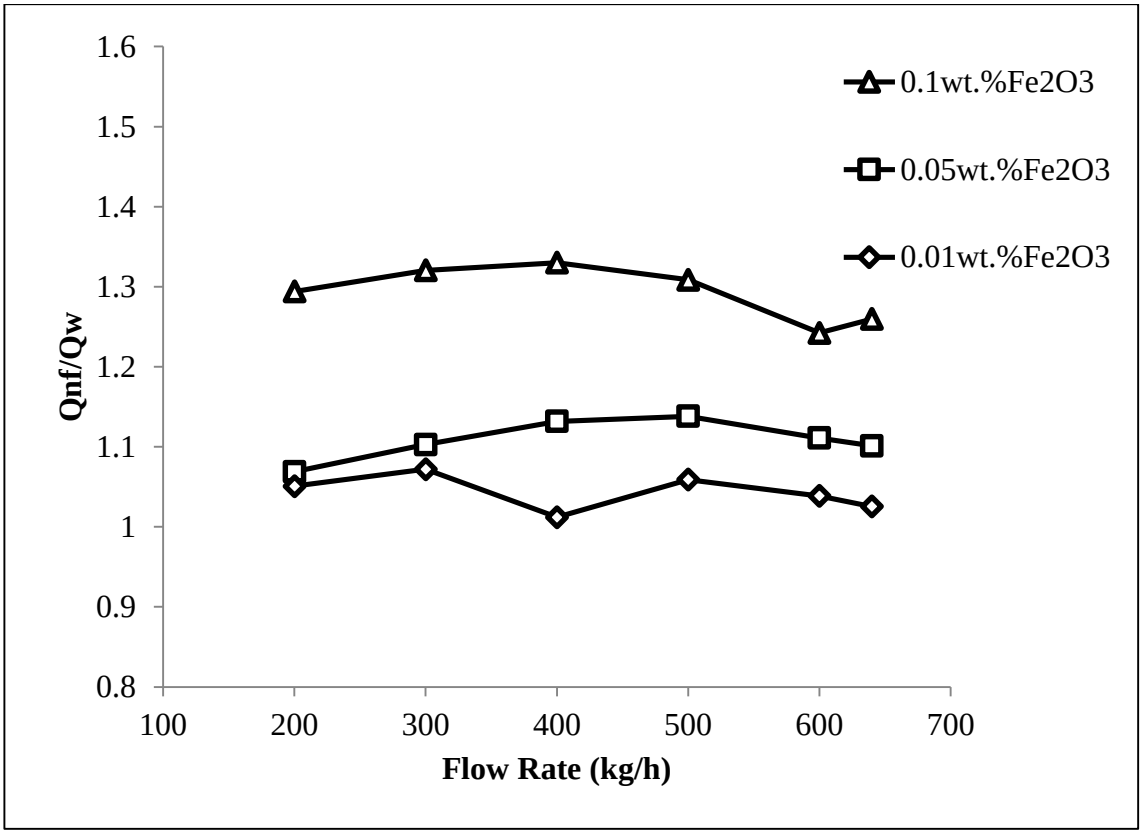

Figure 4:35 Enhanced in heat transfer of CNT- 1 wt%Fe_2O_3 nanofluids as a function of flow rate.

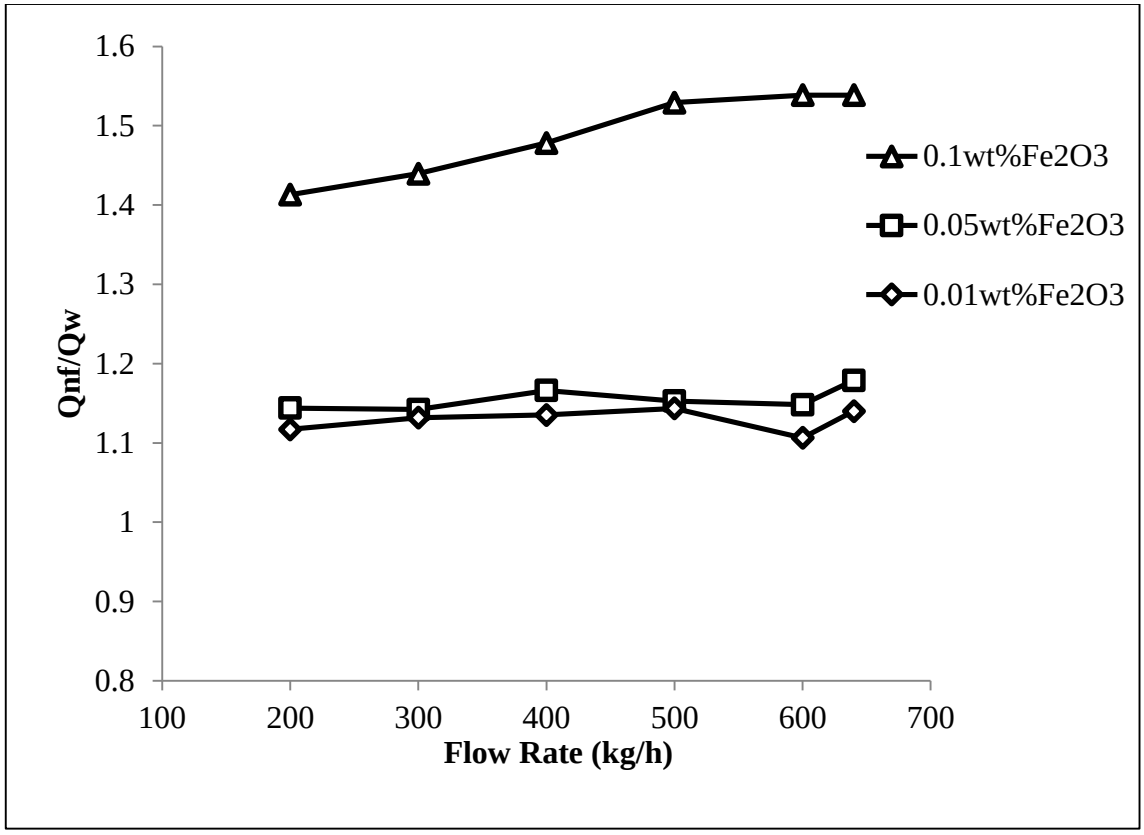

Figure 4:36 Enhanced in heat transfer of CNT- 10 wt%Fe_2O_3 nanofluids as a function of flow rate.

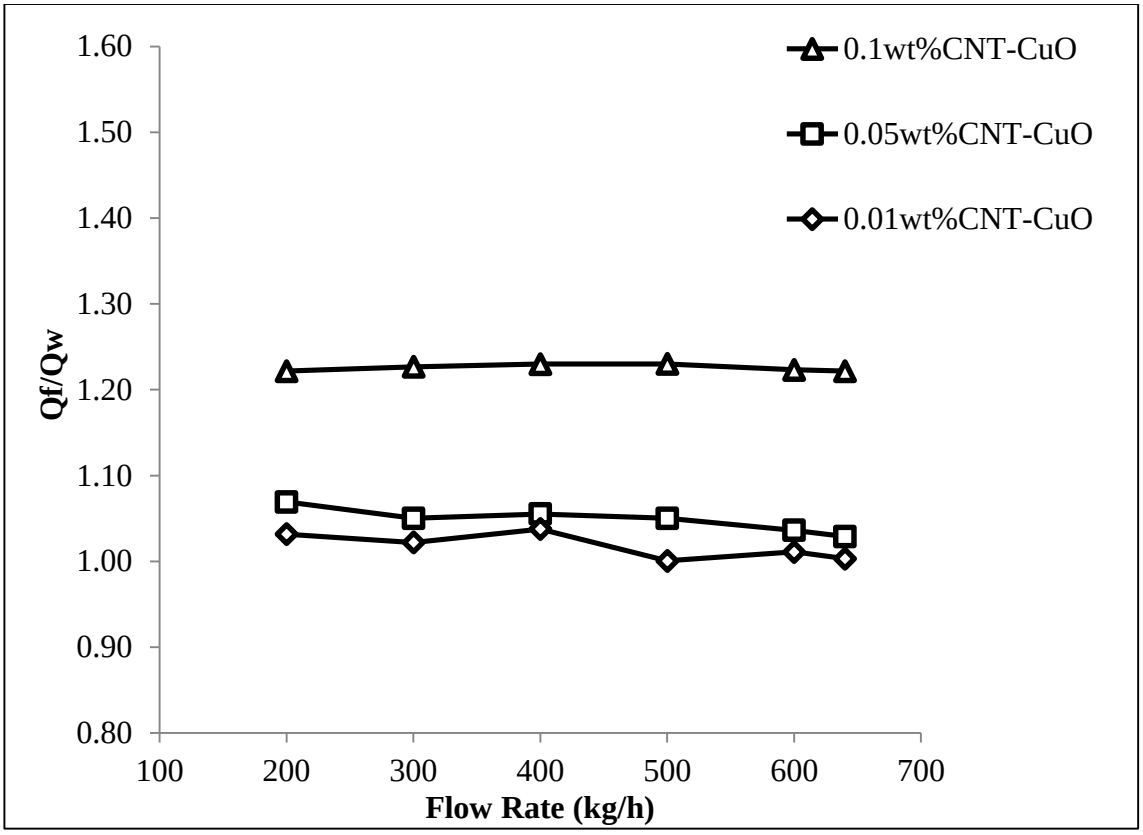

Figure 4:37 Enhanced in heat transfer of CNT-1 wt% CuO nanofluids as a function of flow rate.

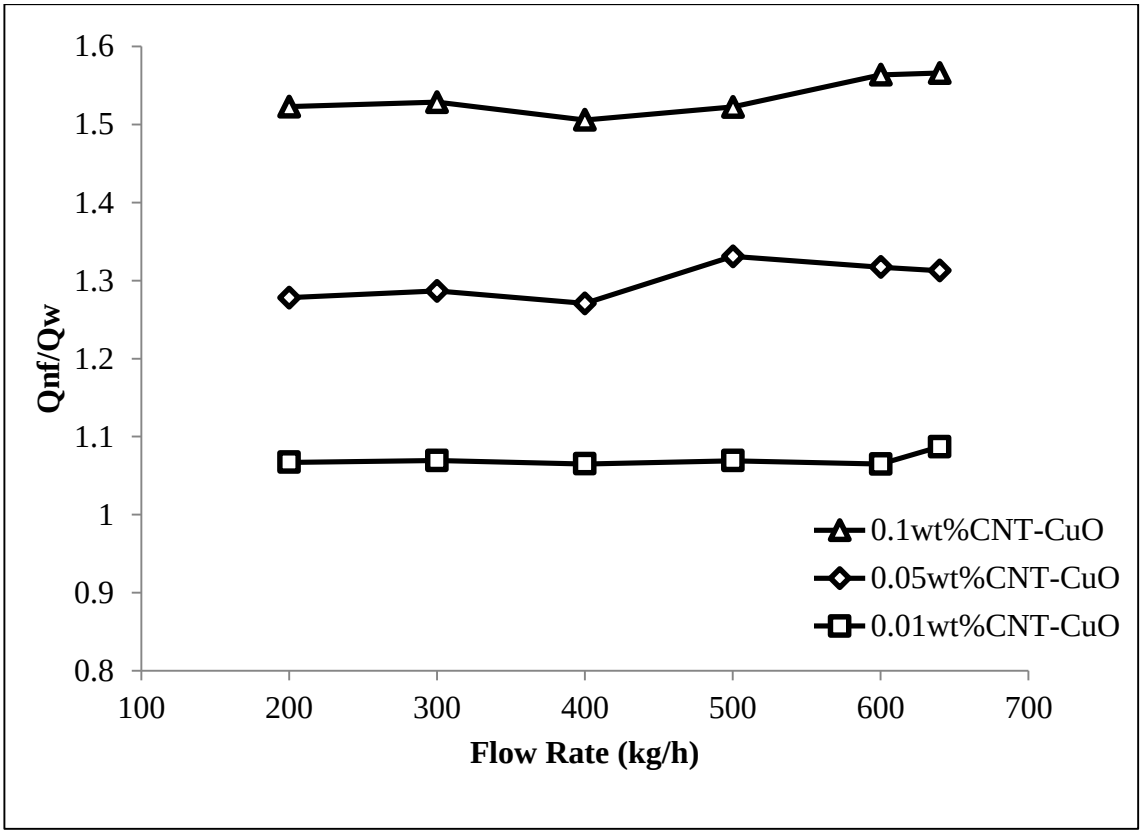

Figure 4:38 Enhanced in heat transfer of CNT-10 wt% CuO nanofluids as a function of flow rate.

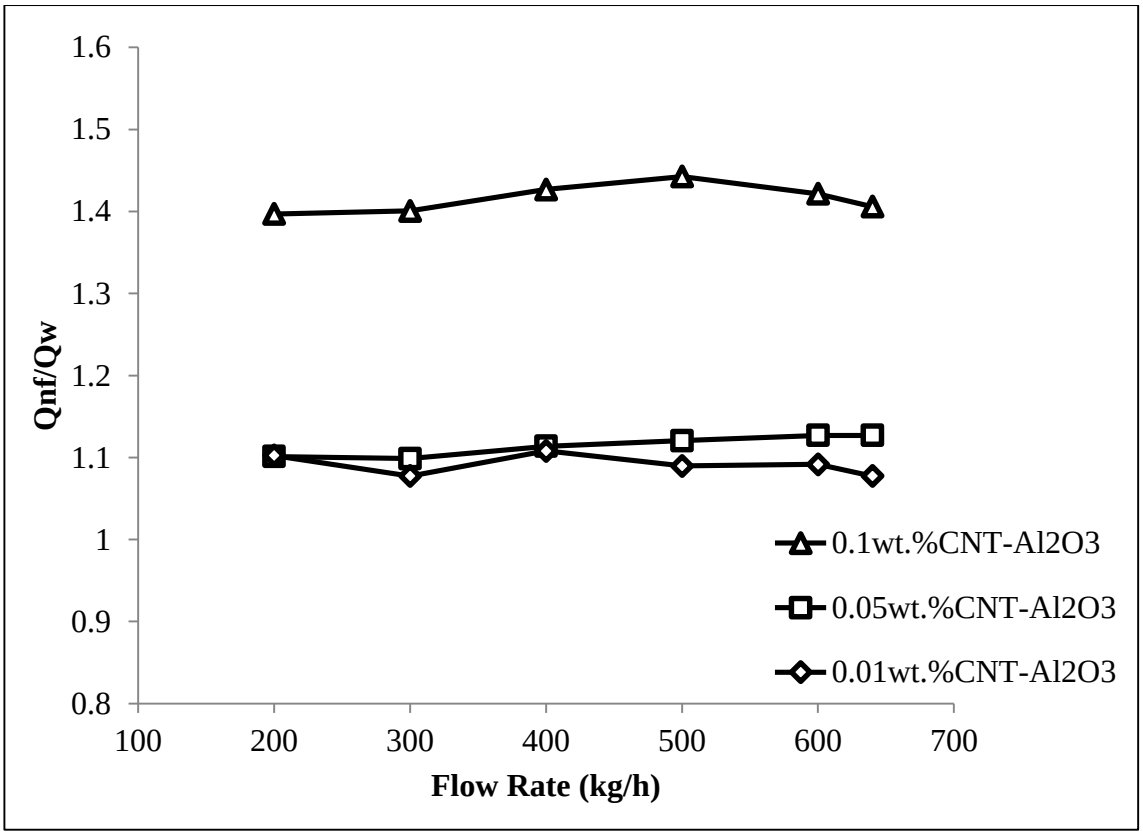

Figure 4:39 Enhanced in heat transfer of CNT- 1 wt%Al_2O_3 nanofluids as a function of flow rate.

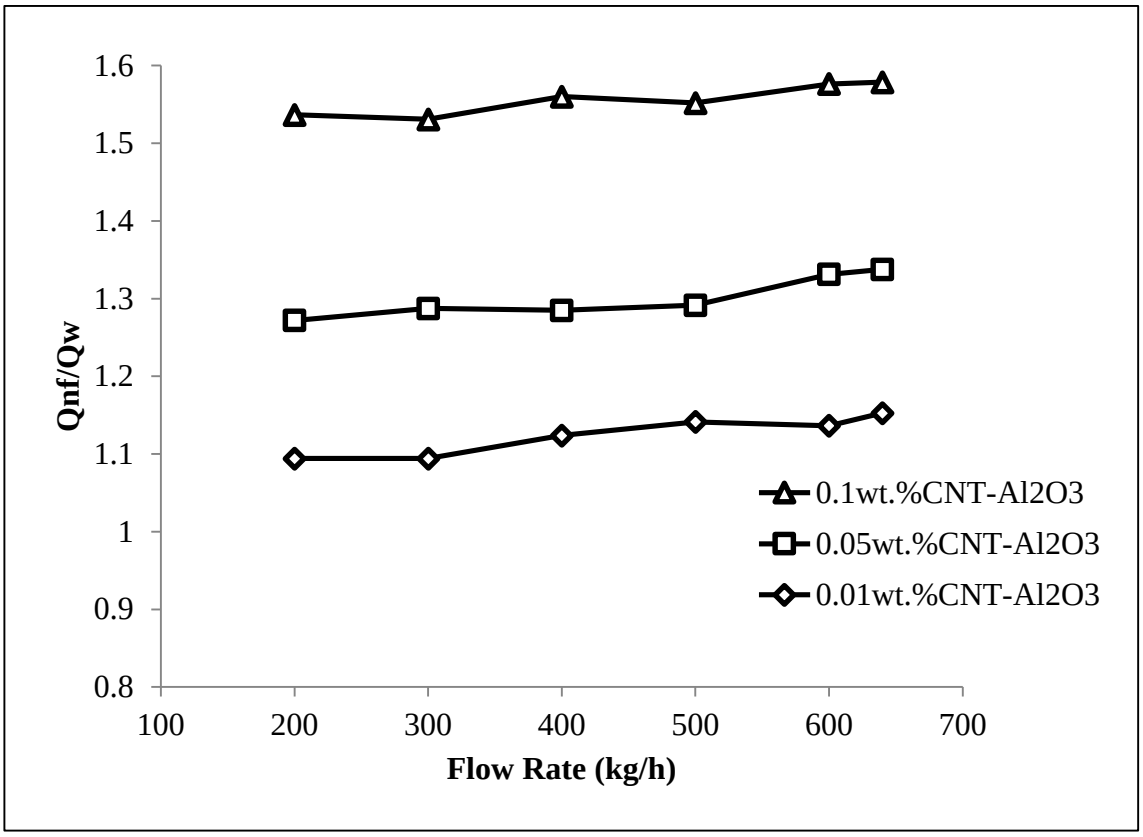

Figure 4:40 Enhanced in heat transfer of CNT-10 wt%Al_2O_3 nanofluids as a function of flow rate.

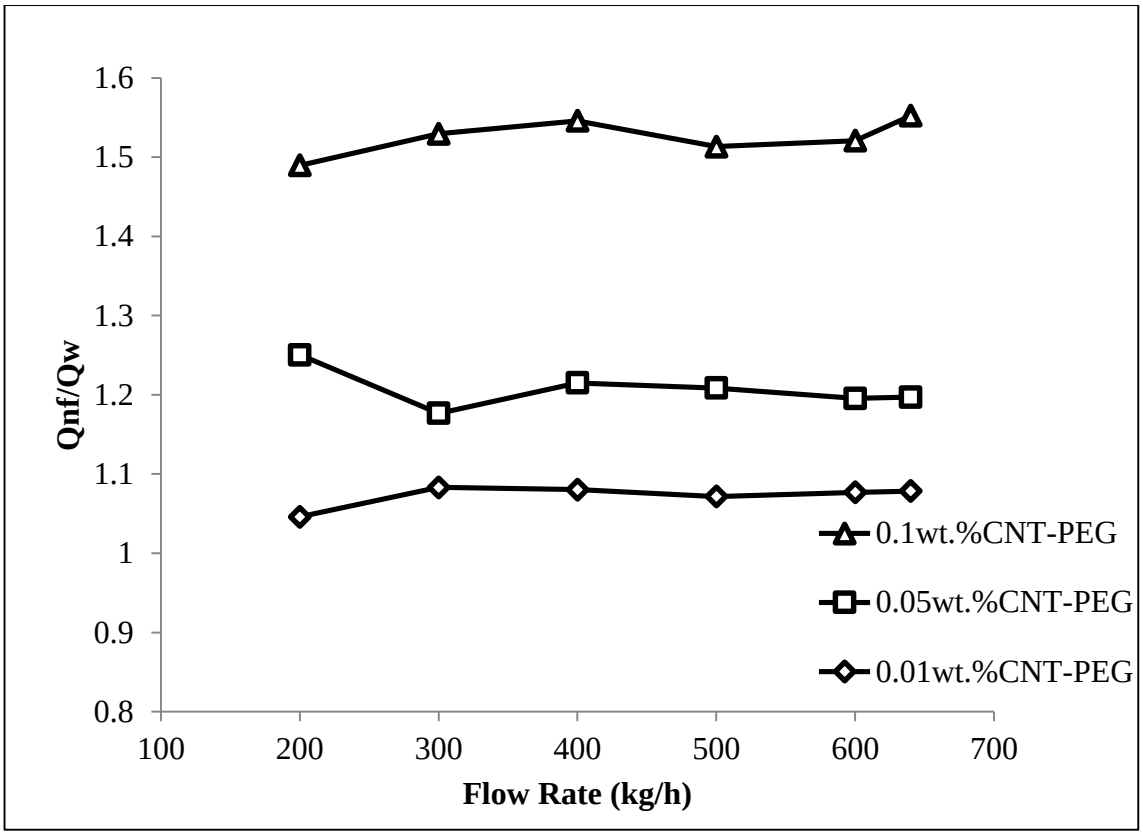

Figure 4:41 Enhanced in heat transfer of CNT-PEG nanofluids as a function of flow rate.

4.7 Pressure Drop of nanofluids

The pressure drop of nanofluids between the inlet and outlet tube was measured using a differential pressure transmitter. The pressure drop was measured for the turbulent flow regime with Reynolds number varying from 5000 to 25,000.

Figures 4.42 through 4.49 show the variation of the pressure drop as a function of the mass flow for unmodified and modified CNTs nanofluid. It is observed that the pressure drop is almost constant and equivalent to water at same condition and at different weight concentration of nanomaterials.

From the previous results of viscosity, it is clear that the viscosity is increased to 3 %, 3.2% and 11% for 0.01% , 0.05 % and 0.1 % weight concentration of CNT nanofluids respectively. An increase in the nanofluids temperature leads to a decrease in the viscosity of the nanofluids, which results in a reduction in the pressure drop.

It is well known that the presence of nanoparticles in the fluid will increase the pressure drop of the system, but in this study it has been noticed that the nanomaterials did not really affect the pressure drop of the system due to the very low surface roughness of the nanomaterials. In addition, the viscosity of modified and unmodified nanofluids decreases at high shear rate due to its shear thinning effect, more basically, arrangement of nanotubes at high shear rates [18,23,24].

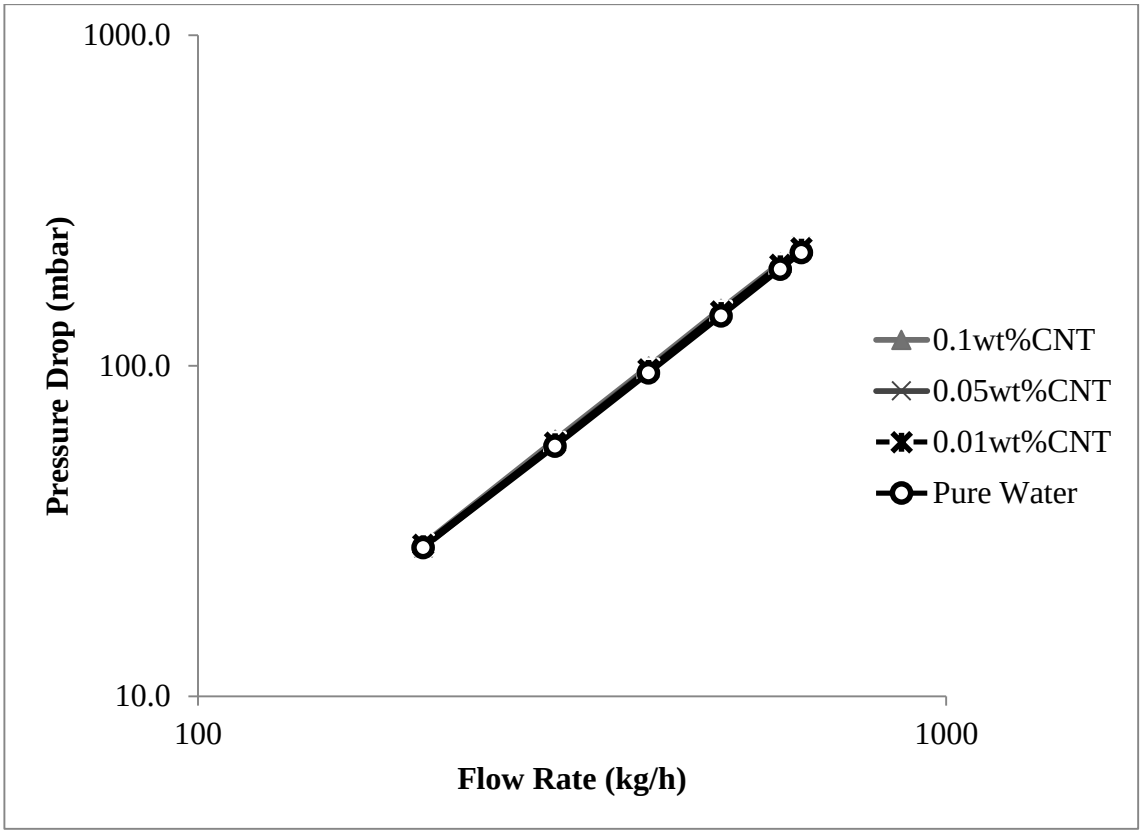

Figure 4:42 Variation in the pressure drop with respect to the mass flow rate for MWCNT nanofluids.

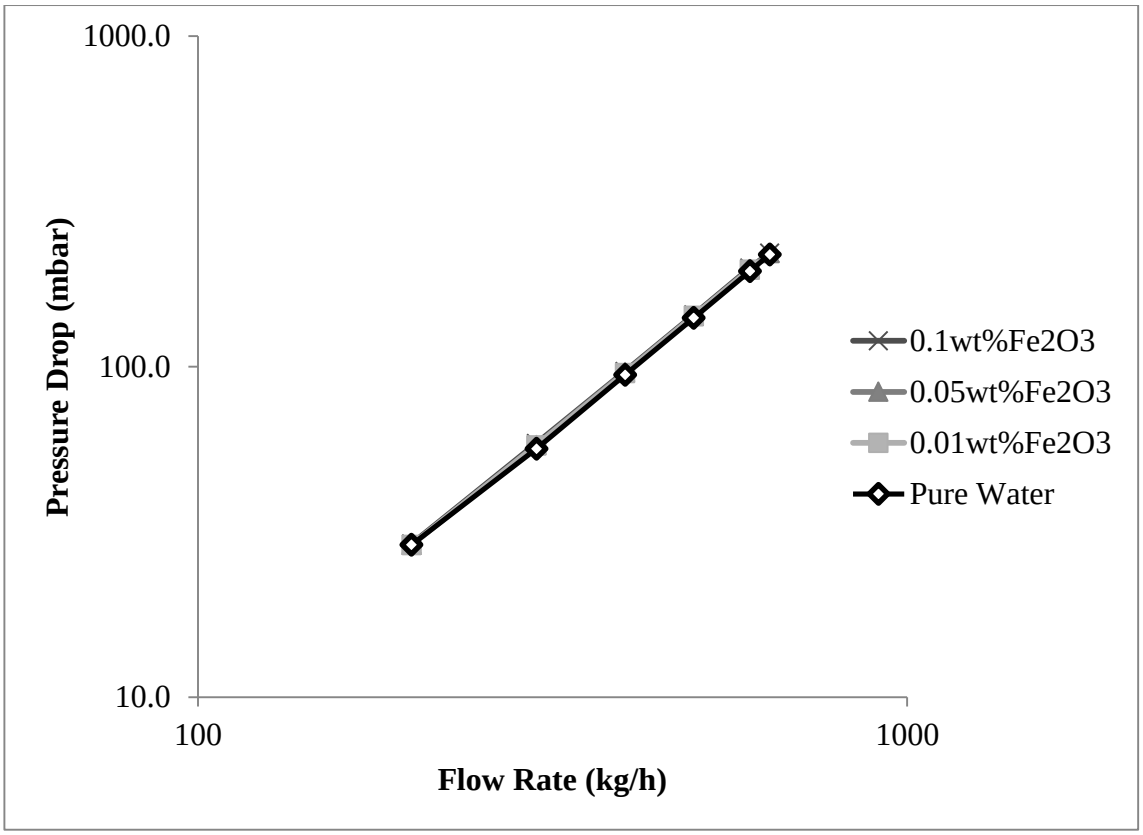

Figure 4:43 Variation in the pressure drop with respect to the mass flow rate for CNT-10 wt%Fe_2O_3 nanofluids.

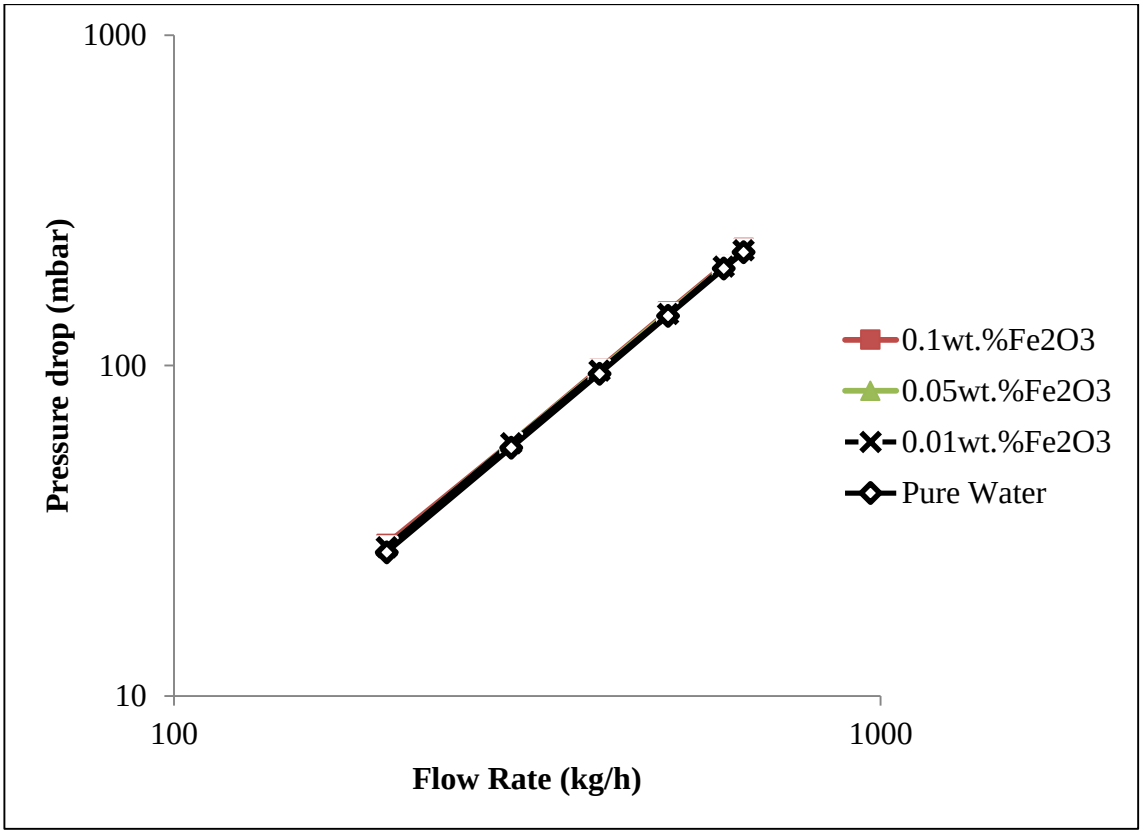

Figure 4:44 Variation in the pressure drop with respect to the mass flow rate for CNT-1 wt%Fe_2O_3 nanofluids.

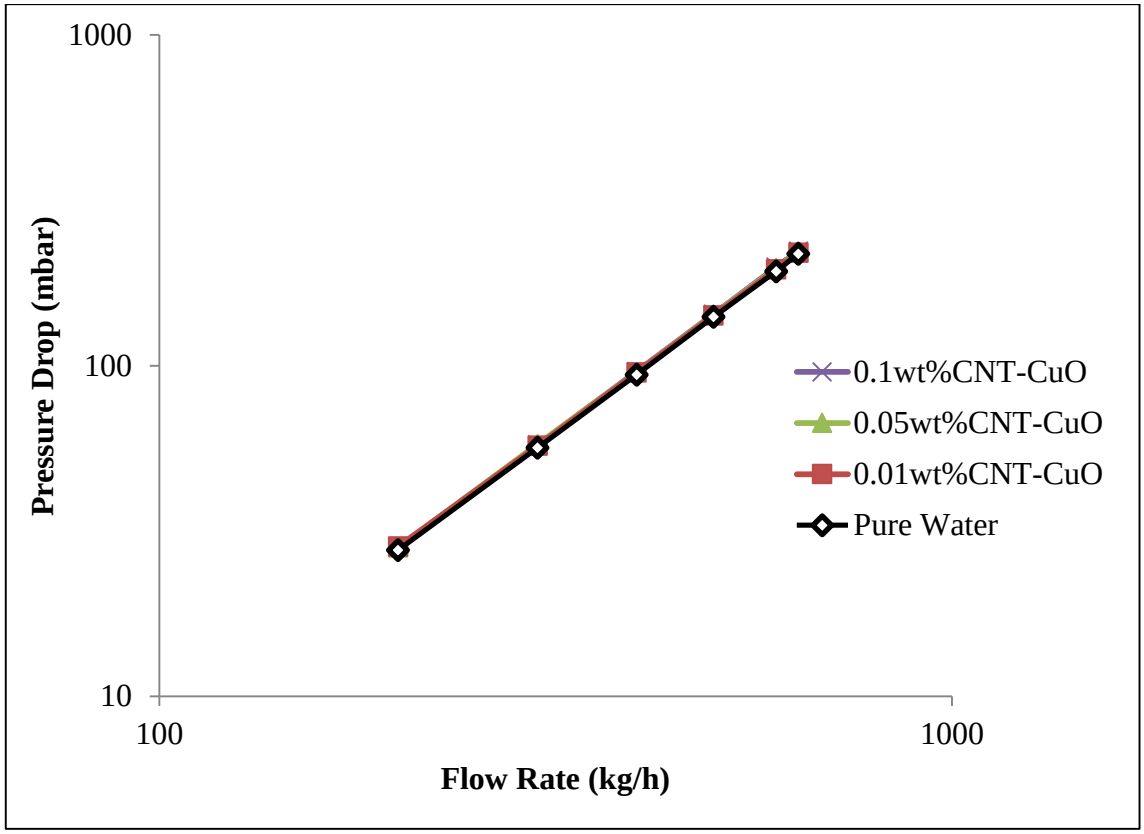

Figure 4:45 Variation in the pressure drop with respect to the mass flow rate for CNT-10 wt%CuO nanofluids.

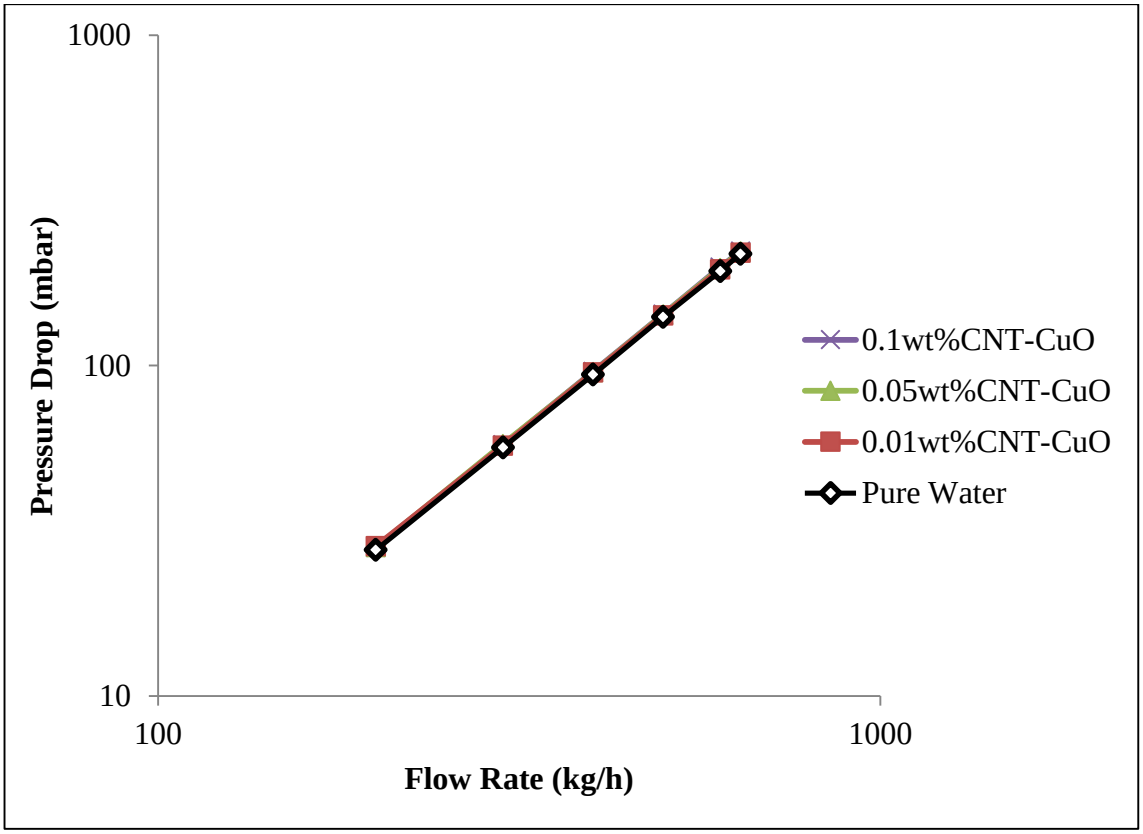

Figure 4:46 Variation in the pressure drop with respect to the mass flow rate for CNT-1 wt%CuO nanofluids.

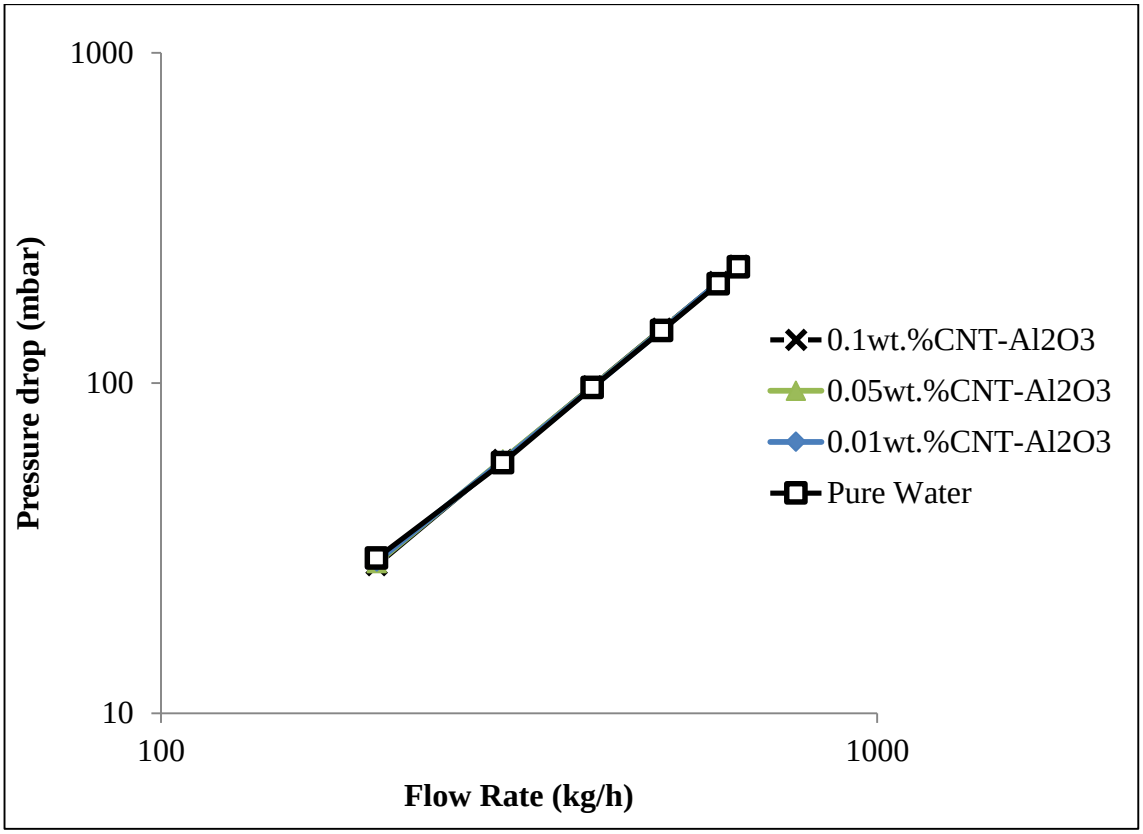

Figure 4:47 Variation in the pressure drop with respect to the mass flow rate for CNT- 10 wt%Al_2O_3 nanofluids.

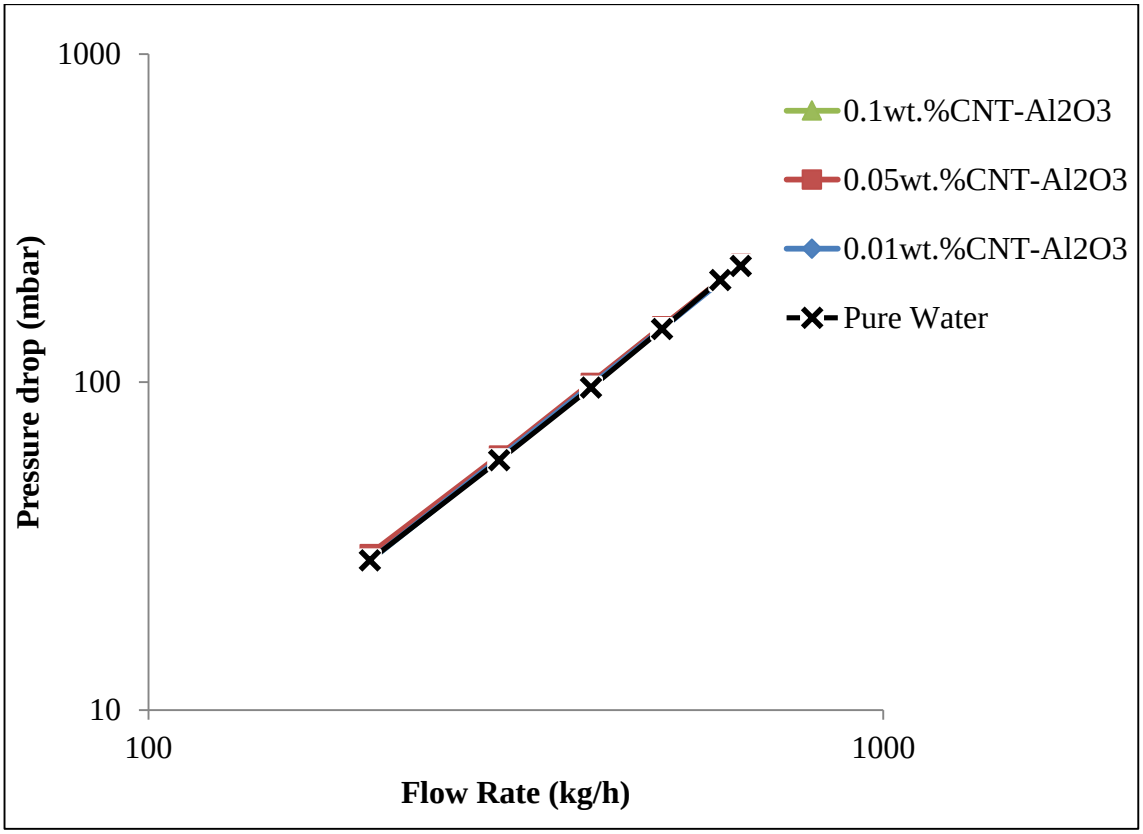

Figure 4:48 Variation in the pressure drop with respect to the mass flow rate for CNT- 1 wt%Al_2O_3 nanofluids.

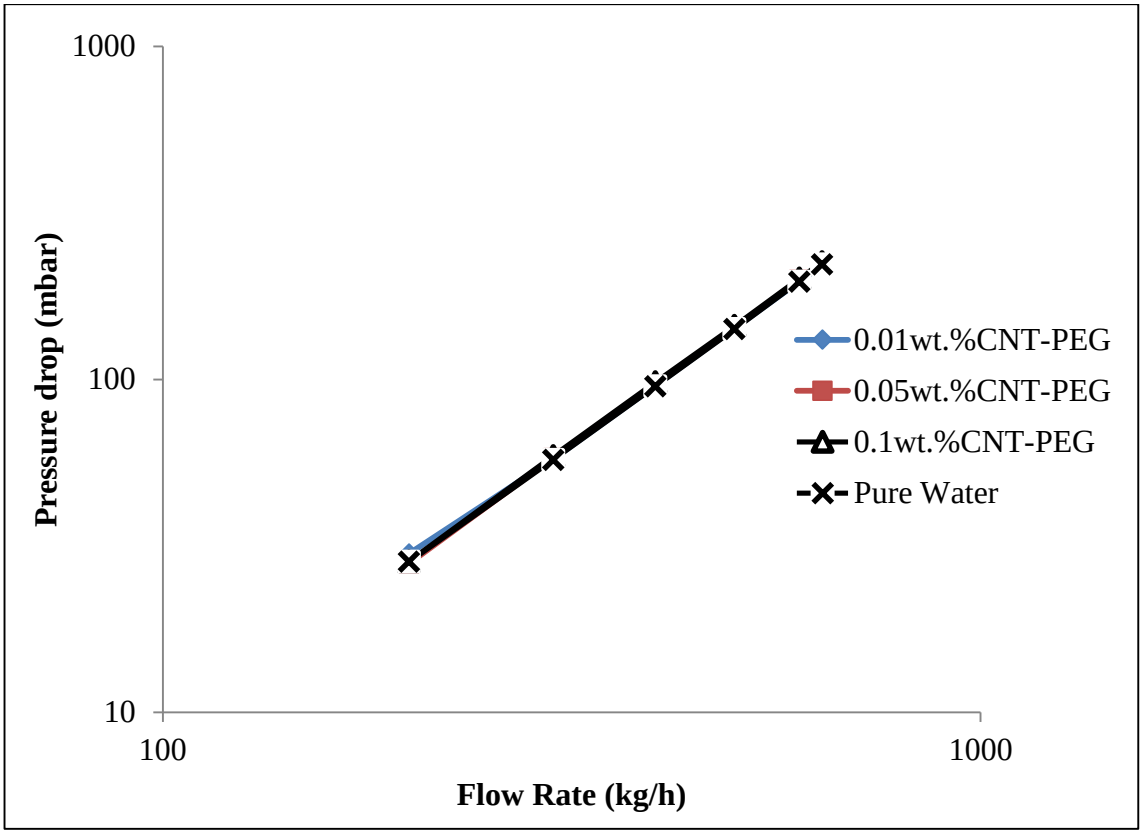

Figure 4:49 Variation in the pressure drop with respect to the mass flow rate for CNT-PEG nanofluids.

4.8 Proposed Mechanism for Enhancement in Heat Transfer

There are many mechanisms that explain the enhancement in the heat transfer of the nanofluid. The first mechanism is the enhancement of the heat capacity of the nanofluid by adding highly conductive nanomaterials such as raw CNTs and modified CNTs with metal oxide nanoparticles. It was noticed that the unmodified CNTs enhanced slightly the heat capacity of the water, while impregnated or modified CNTs with metal oxide nanoparticles increased drastically the heat capacity of water. The second possible mechanism is due to coating of metal oxide nanoparticles on the surfaces of carbon nanotubes which enhanced their separation and reduced the agglomeration. The agglomeration of the nanomaterials is one of the big problems that reduce the heat transfer of the nanofluid. Good dispersion of highly conductive nanomaterials in the fluid will sharply increase the heat of fluid. As explained before, the dispersion of nanomaterials is one of the major factor and in order to obtain a homogenized solution containing well dispersed nanoparticles, two methods could be applied. The first method is ‘physical’ by introducing sonication waves to break down the van der walls interaction forces between the particles. The second method is ‘chemical’ by modifying the surfaces of the particles by adding a surfactant that is soluble in the fluid or adding elements to the surface of particles that reduce their aggregation. In this study, both methods were used, ‘physical’ by using probe sonicator and ‘chemical’ by impregnating/functionalizing the surfaces of the CNTs by metal oxide nanoparticles and PEG, in order to obtain a well dispersed nanofluid. Another possible mechanism is the surface phenomena where the small layer of the unmodified and modified CNTs will coat the wall of the steel tube and enhance the thermal conductivity of the steel.

CHAPTER FIVE

CONCLUSIONS AND RECOMANDATIONS

5.1 Conclusions

The present experimental work was focused on the investigation of thermo physical properties, heat transfer enhancement and pressure drop of the nanofluids with unmodified and modified carbon nanotubes. The surfaces of CNTs were doped with metal oxide nanoparticles using wet impregnation technique and functionalize with polyethylene glycol (PEG) functional groups using fisher esterification process. The CNTs were doped with iron oxide (Fe_2O_3), aluminum oxide (Al_2O_3) and copper oxide (CuO) nanoparticles at two percentage loading (1 and 10%), while PEG functional groups was functionalize based on the percentage of carboxylic functional groups which was used as cross link for PEG with CNTs. The functionalized and impregnated CNTs were analyzed using Fourier Transform Infrared Spectroscopy (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM) and Energy Dispersive X-ray analysis (EDX). Based on the results, the diameters of the CNTs were varied from 20 to 40 nm with an average diameter of 24 nm, while the length of these carbon nanotubes ranged from 10 to 30 μm. In order to verify the presence of nanoparticles ions on the surfaces of the CNTs the back scattered FE-SEM was used. It was observed that, there are many white crystal structures of metal nanoparticles in small sizes and irregular shapes. The distribution and agglomeration of metal oxide nanoparticles was also

observed. At low concentration of metal nanoparticles (1 wt. %), the particles spread widely on the surfaces of carbon nanotubes forming very small crystals particles with diameters varying from 1-5 nm while at high concentration of metal oxide nanoparticles (10 w.t %) the particles were agglomerating forming a big crystals nanoparticles with diameters varying from 1-15 nm. In order to confirm the presence of metal oxide the thermogravimetric analysis (TGA) was used. The degradation process was carried out under air condition, the CNT starts degradation at approximately 550 °C and reached a maximum weight loss of at about 600 °C and completes at about 670°C. However, the CNTs impregnated with 1 wt. % metal nanoparticles started oxidation at 500 °C reached a maximum weight loss of at about 550 °C and completed at about 600°C. While increasing the loading of metal oxide to 10 wt. % shifted the oxidation peak to lower value in which initial oxidation at this condition started at 450 °C with maximum weight losses at 500 °C and completed the oxidation at 540 °C.

The nanofluids were prepared using a two-step method by dispersing nanoparticles into water and applying ultrasonic vibration method for 30 minutes. Different samples of nanofluids were prepared at 0.01wt.% , 0.05wt% and 0.1 wt% nanofluids without using any surfactants or depressants.

The kinematic viscosity of the nanofluids is measured by using Stormer viscometer. The results indicate that the viscosity of nanofluids significantly increased with increasing in the concentration of the CNTs. However, the viscosity decreased as the temperature of the fluid increased. Similar phenomena were observed when CNTs were doped with metal oxides at 1 and 10wt. %. Moreover, similar trend was observed when CNTs was functionalized with PEG. It can be concluded that, there is no effect of the types of

nanoparticles doped on CNTs or the chemical groups functionalized onto the surface of CNTs on the viscosity of the fluid, since the viscosity of the fluid is only a function of the temperature and the particle concentrations.

The specific heat capacity of nanofluids was investigated using DSC technique from 25^{o}C to 50^{o}C at heating rate of 1.5^{o}C/min. The results showed that the heat capacity of nanofluids increased significantly with an increase in the concentration of CNTs. The maximum enhancement of the specific heat for unmodified CNTs was 8%, while for the modified CNTs with 1 wt. % Fe_2O_3 at weight concentration of 0.1 wt.% and 35^{o}C was 19 % and this enhancement was increased to 38 % when increasing the loading of metal oxide. The same phenomena was observed for the doped CNT with aluminum and copper oxide and functionalized with PEG. It can be concluded that the unmodified CNTs enhanced slightly the heat capacity of the water, while modified CNTs increased dramatically the heat capacity of water.

The heat transfer experiments of the nanofluids conducted using single tube heat exchanger. The studies were performed at three weight concentrations of nanomaterial (0.01, 0.05 and 0.1wt. %). The inlet temperature of the nanofluid was at 35 ^{o}C while their flow rate was controlled to be at turbulent regime ranging from 200 -640 kg/h. It was observed that the heat transfer rate increased with an increase in the weight concentrations of unmodified CNTs. The maximum enhancement of heat transfer was 15 % at 0.1 wt % and 400 kg/h mass flow rate. This enhancement significantly increases when impregnated the CNT with metal oxides at 1 and 10% loading. Due to the large enhancement in the heat capacity of the CNTs/ iron oxide composite, the heat transfer of the

nanofluid containing CNTs/ iron oxide composite at 0.1 wt% increased by almost 30 % compared to water as base fluid and 15 % higher than undoped CNTs with water at the same condition. Increasing the loading of the iron oxide nanoparticles on the surfaces of CNTs to 10 wt % increased the heat transfer of the nanofluid up 60 % at the concentration of 0.1 wt%. The same phenomena were observed when doped CNTs with aluminum and copper nanoparticles were used. It could be concluded that the observed enhancement in heat transfer is much higher than that of the increase in heat capacity. However, there is no increase in the pressure drop on the system when these nanoparticles were added to the due to the low roughness of the surface of the nanomaterials.

5.2 Recommendations

Many interesting properties of nanofluids have been reported in this thesis. In the previous studies by other researchers, thermal conductivity has received the maximum attention, but many researchers have recently initiated studies on other heat transfer properties such as heat capacity. The use of nanofluids in a wide variety of applications appears promising. But the development of the field is hindered by (i) the lack of agreement of the results obtained by different researchers; (ii) poor characterization of suspensions; and (iii) lack of theoretical understanding of the mechanisms responsible for changes in properties.

This research work, therefore, concludes by outlining several important issues that should receive greater attention in the near future.

The following recommendations are made to improve the quality of the data and to extend the scope of the research area:

1) Using other types of heat transfer fluids such as ethylene glycol and oil.
2) Carry out the experimental of the heat transfer and the pressure drop of the fluid for long residence time.
3) Use other type of heat exchanger such as U-tube.
4) Use other chemicals such as sodium dodecyl sulphate (SDS) and poly vinyl Pyrrolidon (PVP) to enhance the dispersion of nanofluids.

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